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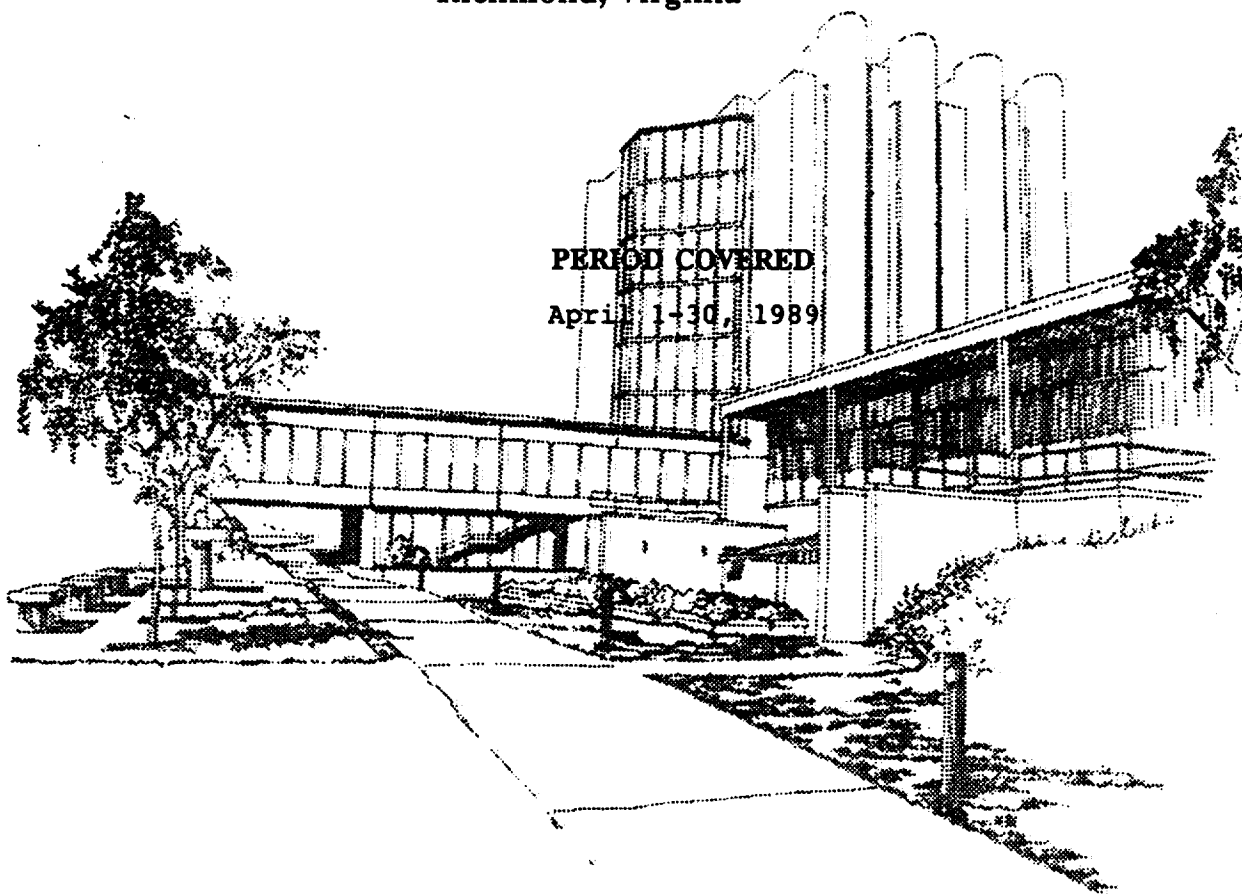
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PHILIP MORRIS USA
Research Center
Richmond, Virginia

PERIOD COVERED

April 1-30, 1989



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*N/R = No Report

PROJECT NUMBER: 2107
PROJECT TITLE: Filter Research & Development
PROJECT LEADER: C. J. Campbell
PERIOD COVERED: April, 1989

I. IMPROVED FILTRATION - INCREASED EFFICIENCY:

- A. **Objective:** Develop filter systems with a higher efficiency than presently available and evaluate them for subjective advantages.
- B. **Status:** Five cigarette models, one using a Celanese CA web dual filter, three using a Filtrona peripheral flow concentric filters, and one control using a 2.6/42,000 tow have been made for Project 605 subjective and POL testing and are now in CI. The RTD of the CA web cigarettes was examined and they were found to have a coefficient of variation of 9%. This compares to a COV of 7.5% on Lark Milds, for example. Variability of tar delivery will be examined when the data is available.

Six models of non-vented 6mg cigarettes using Project 605 filler are now being made in Semiworks. The filters are duals which include versions of CA Web, Concentric with CA Web core, and Filtrona UHF. Analytical and subjective evaluations are planned.

Three models of Merit Ultra Lights using 1.6 dpf tow and a control using 2.6 dpf tow have been made and given to Flavor Development for subjective evaluation to determine the effect of reducing RTD and ventilation while maintaining tar delivery.

II. IMPROVED FILTRATION - MENTHOL STABILITY:

- A. **Objective:** Investigate methods of improving the stability of menthol delivery in smoke of aged cigarettes.
- B. **Status:** Eleven cigarette models, some with heat treated filters, continue in accelerated ageing conditions with periodic evaluation for stability of menthol delivery.

III. IMPROVED FILTRATION - NOVEL FILTER SYSTEMS:

- A. **Objective:** Develop and evaluate new and unique cigarette filters which may offer a distinct product advantage.
- B. **Status:** CONCENTRIC FILTERS: PM Europe is proceeding with plans for market testing a cigarette with a concentric filter in Europe. They have ordered three million filter rods from American Filtrona to begin production.

CARBON FILTER ROD: A comprehensive project status report is being prepared.

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CA PLUGWRAP: Marlboro Lights and Lark Milds KS models were fabricated using a Dexter plugwrap containing a layer of cellulose acetate fibers embedded within the paper structure. This is intended to adhere to the filter tow with triacetin so anchor lines can be eliminated and ventilation variability reduced. Sufficient adhesion was not obtained on the Lark due to the short filter segment involved. The Marlboro Lights model is now being examined. Further work is planned to attempt to increase the adhesion.

IV. IMPROVED FILTRATION - SELECTIVE FILTRATION:

- A. Objective: Explore the use of specific additives in filters for selective filtration or subjective modification of smoke.
- B. Status: FML: A report summarizing the initial work done with additives and their ability to improve the subjective character of the smoke compared to untreated FML filters has been issued. Additional work with surface additives is continuing to attempt to achieve parity with a CA filter.

ART: Plans are being made to fabricate fluted recessed filters to test their ability to enhance subjective impact. Also, carbon treated with acidic or basic additives will be placed into filters to provide selective filtration and modify the subjective response. Prior art of selective filtration has been reviewed by Physical Research and selected methods will be incorporated into filter models for evaluation on removal of gas phase components.

V. FILTER SUPPORT FOR OTHER R&D PROGRAMS:

- A. Objective: Provide design assistance and potential new filter systems for other R&D programs.
- B. Status: CARBOWAX REPLACEMENT: Models of three types of cigarettes with charcoal filters have been made with triacetin plasticizer and evaluated versus Carbowax controls for gas phase filtration after ageing. Analytical results show that gas phase removal is only slightly diminished over time with the triacetin filters. Subjective evaluations by the Richmond Panel of these models have not indicated a clear preference for any one model.

An ageing study now in progress shows that carbon removes triacetin from CA filters over time. The current analytical technique does not detect this absorbed triacetin.

A test is being planned to evaluate the performance of both Carbowax and triacetin filters which are aged without being attached to cigarette rods in an effort to isolate the effects the tobacco may have on the active carbon. These will be attached to tobacco rods with no dilution just prior to smoking and gas phase reduction will be measured.

LARK SUPER LIGHTS: Models of Lark Super Lights cigarettes which achieve a reduced tar delivery of 7mg from the current 9mg without increasing ventilation or RTD were successfully made using 1.6/41,000 tow. Sample 27mm plug-space-plug filters submitted by Filtrona England are currently being evaluated.

LARK VENTILATION VARIABILITY STUDY: Analytical results of seven test models of Lark Milds cigarettes made in the Manufacturing Center with various combining paper types are promising. Three of the models, one with a Filtrona MPF filter, and two with a patterned hot melt on the combining wrap, show lower ventilation variability than is normally seen on Lark. All three of these also machined well.

Additional models using inherently porous combining papers such as 70-S1 were attempted to be made April 22, however the run was unsuccessful because the adhesive system was not able to adhere the components to the combining paper. An alternate glue system is being developed and the test will be repeated.

Semiworks has completed setting up a Hauni Laser on one of their tippers for on line perforation. Cigarette models are scheduled to be produced the week of May 8. These will then be evaluated for ventilation variability. An initial cost estimate for converting Lark to a Hauni on line Laser system has been prepared by Industrial Engineering at the MC and has been issued.

Information on a variety of plug-space-plug filters from competitive brands has been gathered to access the ventilation variability of other types of construction. The analysis supports our current development ideas.

Dexter is submitting sample bobbins of their "tea bag" material which is a porous paper with polypropylene fibers on one side. These fibers are heat sealable, but retain the inherent porosity of the paper. This material will be tested in place of the current mechanically perforated combining paper.

PROJECT NUMBER: 2108
PROJECT TITLE: New Product Technology
PROJECT LEADER: W. T. Callahan
WRITTEN BY: W. A. Geiszler
PERIOD COVERED: April, 1989

I. SAUNA

- A. Objective: Develop a dual charcoal filter as a Sauna-type filter system for a Japanese product.
- B. Status: Further modifications are being made to the DATA filter slitter to reduce the depth of cut into the filter. Combined charcoal plastic fluted filter rods are available to run on the slitter.
- C. Plans: (1) Slit filter rods and produce cigarettes with blocked flutes for smoking analyses. (2) Evaluate Sauna designs that do not require hot melt.

II. KAYMICH MENTHOLATED CIGARETTES

- A. Objective: Evaluate cigarettes mentholated by the Kaymich applicator system.
- B. Status: A test was conducted to determine menthol losses from cigarettes mentholated via the wrapper prior to packaging. The menthol level dropped from 4.4 to 3.8 mg during one hour of cigarette exposure and continued at that rate of loss through 3 hours of exposure. Variation in cigarette hold time between making and packing makes this loss rate unacceptable.

A bobbin of foil was mentholated by Process Development using the Kaymich system to apply molten menthol. The foil will be compared to conventional solution-mentholated foil.
- C. Plans: (1) Evaluate cigarettes packed with Kaymich mentholated foil. (2) Determine menthol loss from cigarettes made with Kaymich application of menthol to filler on the maker.

III. AMBROSIA:

- A. Objective: Evaluate flavor-release compounds applied to cigarette wrapper.
- B. Status: Both direct application of flavor-release compounds to the wrapper and incorporation into the sideseam adhesive are being explored. Two materials were coated on paper using a pipe coating technique, and cigarettes have been produced for evaluation. Several adhesive formulations containing flavor-release agents were also run on the maker to produce cigarette samples.

- C. Plans: Evaluate cigarettes containing flavor-release compounds and produce further samples as required.

IV. EMBOSSING TECHNOLOGY:

- A. Objective: Explore embossing technology for potential new product development.
- B. Status: A torque control system was installed on the laboratory embossing machine which greatly facilitates the operation. Multiple bobbins of edge-calendared two ply cigarette wrapper were produced for Trim V testing.
- C. Plans: Produce bobbins of intermittent calendared wrappers for evaluation on cigarettes.

PROJECT NUMBER: 2304
PROJECT TITLE: Flavor Development/Domestic Product Development/Technology Support
PROJECT LEADER: G. N. Yatrakis
PERIOD COVERED: April, 1989

I. MENTHOL RELEASE COMPOUNDS:

- A. Objective: Develop a mentholated charcoal filtered cigarette utilizing a menthol release compound. Also, to apply menthol release technology to other areas.
- B. Status: Charcoal filtered cigarettes have been produced with GMC sprayed on filler and sprayed plus additional GMC via the "Ink Jet Injector". Menthol-in-smoke values of 0.21, 0.24, 0.29 and 0.31 mg of menthol per cigarette were achieved.
- C. Plans: Optimize procedure and produce prototypes for subjective panel testing.

See New Product Development, 2304, 4015 for cooperative projects.

PROJECT NUMBER: 2306
PROJECT TITLE: Marlboro Standardization and International Support
PROJECT LEADER: J. L. Spruill
PERIOD COVERED: April, 1989

I. MARLBORO STANDARDIZATION

- A. **Objective:** Analytical and subjective evaluations of production Marlboro KS/LS.
- B. **Status:** March 7, 1989 factory pick-ups complete. Standard VII Run is scheduled for week of July 17 at all factory locations. Preliminary meetings are in progress. Factory pick-ups of Marlboro Lights received for April 17.
- C. **Plans:** Evaluations of Marlboro Lights pick-ups.

II. DOMESTIC CIGARETTE DEVELOPMENT PANEL

- A. **Objective:** To provide subjective direction for programs within R&D and manufacturing locations.
- B. **Status:** Fifteen panels completed for the reporting period in addition to evaluations of production Marlboro Ultra Lights from Cabarrus and ten brand profiles.
- C. **Plans:** Provide assistance as needed.

III. PROJECT NATURAL

- A. **Objective:** To develop 85mm and 100mm full-flavored and lights prototypes using blend components and flavor systems which will result in a natural blended product.
- B. **Status:** Blend E models (flavored and unflavored) were evaluated. Blend C (no flavor additives) processed in Semi-works and made at M/C for B&H 83mm Box product. Requests submitted using "natural" flavors on current Marlboro blend.
- C. **Plans:** Flavor compounding for current requests.

IV. FLAVOR RESOURCE DATA BANK

- A. **Objective:** Creation, customization and maintenance of flavor resource and data files for use by Flavor Development Division.
- B. **Status:** New vendor samples continue to be evaluated subjectively.
- C. **Plans:** Division updated with information.

V. INTERNATIONAL SUPPORT**International Brands Smoking Panel**

- A. **Objective:** Subjective evaluations (rod aroma and smoking characteristics) of cigarette brands in the international market.
- B. **Status:** Twelve panels completed during the reporting period as well as three brand profiles and evaluations of Parliament Lights King Size and 100 box.

PROJECT ULTRA-JAPAN

- A. **Objective:** Explore the use of new blends, new flavor systems and different construction styles. Cigarettes will be in the low and ultra-low category for the Japanese market.
- B. **Status:** Six total blend casing models received for subjective evaluations and aftercut development.
- C. **Plans:** Complete subjective evaluations. Flavor development in progress.

PROJECT OLYMPIC (KOREA) - BRONZE

- A. **Objective:** Development of a product to be competitive to Pine Tree King Size.
- B. **Status:** Casing and aftercuts completed for primary processing.
- C. **Plans:** Evaluations of prototypes to be made.

PROJECT OLYMPIC (KOREA) - GOLD/PMSL GOLD

- A. **Objective:** Development of a Virginia sweet product for the Korean market.
- B. **Status:** Casings and aftercut supplied for primary processing.
- C. **Plans:** Evaluations of prototypes to be made.

PAN ASIAN MENTHOL - PROJECT CEDAR

- A. **Objective:** Development of free-standing menthol model to compete with Salem Lights (Hong Kong).
- B. **Status:** Samples made did not meet menthol targets. Remake is being scheduled for large scale using two blends and several flavor systems. Models will be made using flush and recessed filters.
- C. **Plans:** Evaluations of models to be made.

MERIT II (HONG KONG)

- A. Objective: Development of a product to be competitive with Kent.
- B. Status: Awaiting completion of six prototypes from make-pack.
- C. Plans: Evaluations of models, analytically and subjectively.

MARLBORO IMPROVEMENT PROGRAM (AUSTRALIA)

- A. Objective: Develop an Australian Marlboro subjectively closer to the U.S. Marlboro.
- B. Status: Cigarette models complete and under subjective and analytical evaluations.
- C. Plans: Complete evaluations.

MEXICO

- A. Objective: Modifications of the existing Marlboro flavor system to be brought in line with U.S.
- B. Status: Consumer tests still in progress.

PROJECT NUMBER: 2307
PROJECT TITLE: Flavor Investigation/Processed Tobacco
PROJECT LEADER: R. W. Hale/J. Swain
PERIOD COVERED: April, 1989

I. FLAVOR INVESTIGATION:

A. Objective: To provide analytical support for activities related to development and application of flavoring materials.

B. Results:

1. **Low Tar/High Flavor:** Method development was started on total smoke isolation, fractionation and GC/MS Analysis. These methods will be used to establish a reliable qualitative and quantitative comparison of smoke composition for cigarettes with the same filler but with different degrees of ventilation.
2. **Glycerin/Triacetin:** Extractions of the impurities from both glycerin and triacetin have been completed. The quantities of the impurities are now of sufficient amounts so that identification can be completed by GC/MS. Models utilizing natural/synthetic blends of triacetin on the filter plugs are being subjectively evaluated this week.
3. **Operations Support:** Analyses of anethole on 36 filler samples have been completed for the total blend silo and A/C cylinder capacity test for the Manufacturing Center. Analyses of anethole on 50 filler samples have been completed for the A/C application optimization study in Semi-works. Seven samples of Marlboro 85 cigarettes, March 7, 1989 factory pick-up, were analyzed for anethole.
4. **Miscellaneous Internal Analytical Support:** Ethanol, PG and water were determined in 19 aftercut samples for different projects. GC profiles and GC/MS identifications have been completed for 10 natural oils. Headspace volatiles were run on several cigarette samples.

II. PROCESSED TOBACCO

A. Objective: To develop basic and applied knowledge for the purpose of improvement or selective modification of subjective properties of processed tobaccos.

B. Results:

1. **ART Stem Utilization:** Marlboro blends are being evaluated with pilot RL's incorporating slightly less than 2% ART stem in combination with RCB containing 11% ART stem. Leaf Department blend projections based on these levels of usage in sheet materials would account for less than 50% utilization of

ART stem. To allow higher usage in RL's, post-treatments are being tested. Initial results of Hauni tunnel steam treatment of wet-ART stem has shown promise compared to untreated dry-ART stem at a higher level (7.2%) in RL's. Intermediate levels of substitution being made in RL pilot plant this week.

Internal panel tests of ART stem increased to 22% replacing burley stem in RCB have shown subjective differences in Marlboro blends. Intermediate level of 16.5% usage to be evaluated by blending previous test RCB's (50/50) to determine subjective threshold prior to further production RCB trials.

2. Dry Flavor Replacement: Discussions with vendors of roasted JONEX will proceed with increased emphasis on reducing batch-to-batch variability. Larger scale samples to be requested to confirm analytical specifications and subjective parity.
3. ASTA Sheet: Remake of Spanish Marlboro cigarettes with 7% ASTA replacing RCB showed more typical deliveries. Subjective evaluations in progress.

PROJECT NUMBER: 4009
PROJECT TITLE: Development Smoke Studies
PROJECT LEADER: B. L. Goodman
PERIOD COVERED: April, 1989

I. Reduced Sidestream Cigarettes:

- A. Objective: Develop subjectively acceptable cigarettes with reduced sidestream visibility.

B. Status:

Trim V: Additional papers were received from Kimberly-Clark in a continuous effort to determine acceptable limits for additive levels, both from a subjective standpoint and for sidestream visibility reduction. The new series consisted of six different succinate levels ranging from 2.0% to 7.6%. The cigarette showed the expected variation in puff count and sidestream visibility, but the visibility reduction was acceptable (at least 70%) for all six models. The highest level was unacceptable from a subjective standpoint. The lowest level gave a bland taste relative to the high succinate model. Screening of the other models as well as analysis of the papers are in progress.

Bilayer papers made at the University of Maine with the same total composition as the laminated Trim V system were evaluated. One bilayer paper was made to 16 Coresta, while the other one was nonporous, and later perforated to 45 Coresta. A composite sheet with all the additives mixed together was also perforated. The three different papers performed equally well subjectively and gave similar sidestream reductions. The double wrap system from K-C gave slightly higher reduction and a better looking ash. The composite sheet burned faster than the other models, but gave similar deliveries.

Trim V cigarettes were taken to the M/C Panel for subjective comparison to a modified blend. The modified blend utilizes #8 Bright, DBC Burley, MT Oriental, and RLTC. There were no differences in mainstream deliveries or in sidestream visibility.

Lotus: A control Marlboro Lights 100 was tested on the M/C Panel for comparison with previously tested 100 mm low sidestream cigarettes made to a 24.0 circumference with the 2I low sidestream blend and 35% Mg(OH)₂ paper. The experimental models were made with a charcoal and a regular CA filter. Results showed no significant differences in acceptability between the cigarettes. The only significant difference was that the charcoal model gave higher off-taste. Acceptability scores ranged from 4.07 to 4.23.

Standard circumference cigarettes were also made with 35% Mg(OH)₂ paper, with and without different innerliners. Panelists could tell a subjective difference between single and double wraps, but there was no definite preference for either system. The double

wrap models gave a more flaky ash, and increased the visibility reduction.

Results have been received for the cigarettes made for department wide mainstream and sidestream analysis and subjective evaluation. A conventional paper, a 35% Mg(OH)₂ paper, and the K-C double wrap system gave different burn rates, with tar per puff ranging from 0.92 to 1.17 mg. Marlboro and the 2I blends were used with each paper. Subjective screening indicated that all six models had different character, and will be more extensively evaluated by PED. Weight selection is in progress, and the cigarettes will be canned for future use.

II. Sidestream Visibility Measurements:

- A. Objective: Determine sidestream visibility and evaluate methods of analysis.
- B. Status: The eight port visibility instrument was used for a number of visibility measurements. Several changes were made to improve the running. Machined holders with the right dimensions for ultraslim cigarettes were received, and a new, higher capacity pumping system was installed for exhausting the smoke.

A protocol was set up for qualifying the instrument through U.S. Testing. In the first round of testing, three cigarette models of 100 mm length and standard circumference will be used; a control conventional cigarette (Marlboro Lights), a low sidestream model (35% Mg(OH)₂ paper), and one model giving visibility between the two extremes (single wrap K-C paper). Each model will be burned twice a day for five nonconsecutive days, after which the data will be sent out for analysis.

III. Tipping Paper Investigations:

- A. Objective: Develop cigarettes with reduced levels of filter flare-up and coal drop-off.
- B. Status: Four bobbins of iron oxide colored tipping paper were received from Ecusta: one control bobbin colored with standard iron oxide; one bobbin colored with food grade iron oxide; one bobbin colored with normal iron oxide and containing particulate magnesium hydroxide; and one bobbin colored with normal iron oxide and containing precipitated magnesium hydroxide.

Cigarettes were made in a Marlboro K.S. configuration using the four experimental tipping papers as well as production Marlboro tipping. The cigarettes were tested for filter flare-up and coal drop-off. All of the models made had 0% filter flare-up. Cigarettes made with production tipping paper had 2% coal drop-off; cigarettes made with the Ecusta control tipping paper colored with standard iron oxide had a 50% level of coal drop-off; those made with cork tipping colored with food grade iron oxide had 28% coal drop-off; cigarettes made with the tipping containing

particulate $\text{Mg}(\text{OH})_2$, had 0% coal drop-off; and the ones made with tipping containing precipitated $\text{Mg}(\text{OH})_2$, had a 1% level of coal drop-off.

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PROJECT NUMBER: 2304, 4015
PROJECT TITLE: Domestic Product Development
PROJECT LEADER: G. N. Yatrakis, J. B. Easley, and L. S. Wu
PERIOD COVERED: April, 1989
WRITTEN BY: L. S. Wu

I. LOW TAR/HIGH FLAVOR:

A. MARLBORO ULTRA LIGHTS:

1. **Objective:** To develop 85/100 mm Ultra Low (6 mg) candidates for Marlboro line extensions.
2. **Status:** Test market started in Indianapolis, Indiana with blue packs and white tipping and Portland, Oregon with red packs and cork-on-white tipping on May 1, 1989 in king size and 100mm versions. Sample sixes were produced at Stockton Street during the month of April.

3. **Plans:**

Test Market

May 1, 1989

B. PROJECT EXTRA:

1. **Objective:** The development of 4-8 mg product candidates that have the taste of products with twice the tar.
2. **Status:** Filler has been made for both the low density design study at 6 mg and the sidestream study. The low density cigarettes for the design study were made this week and sidestream cigarettes will be made by the second week of May. The low density sidestream study cigarettes utilize Sol-gel coated paper to control puff count. All the Control regular density cigarettes for both studies are in C.I.

3. **Plans:**

Evaluate alternate filters
Low density rod program

On-going
On-going

C. PROJECT 605:

1. **Objective:** To develop a 6mg free-standing cigarette which appeals to flavor low smokers.
2. **Status:** Cigarettes have been made with Blend A and CA, CA web/CA and peripheral flow concentric filters for internal testing. Cigarettes are in analytical.

3. **Plans:**

Filter Evaluations

On-going

D. MERIT SUPER LIGHTS:

1. **Objectives:** To develop a 2mg "Merit-type" cigarette.
2. **Status:** Limited subjective evaluation of models comparing CA, CA-web, CA-1.6dpf, and Paper/CA filters indicate a slight preference for the CA-1.6dpf model. However, this model did not meet tar delivery. The above models contained Total Blend Casing 7695-163F, Aftercut 7695-178E and 50% partially expanded burley in the blend.

Additional models are being subjectively evaluated which contain 35% partially expanded burley and 15% partially expanded bright in CA and paper/CA filter configurations.

3. **Plans:**

POL test Production
Prototype Evaluations

2nd Qtr.'89
Ongoing

E. NOVENT:

1. **Objective:** Develop a 6 mg tar cigarette without filter ventilation.
2. **Status:** Prototypes have been requested using 605 blend A and 100% XTH blend, both to be made with CA and CA-web filters. These models will be used to determine future blend modifications.

3. **Plans:**

Prototype evaluations

On-going

II. TRIM I:

- A. **Objective:** To develop ultraslim product candidates with 17mm circumference that demonstrate product advantages over Capri and Capri Menthol.
- B. **Status:** POL 7182 (Model 2 Regular vs. Capri) was shipped April 24.
- C. **Plans:**

POL Testing
Test Market

2nd Qtr.'89
TBD

III. LOW SIDESTREAM:

A. TRIM V:

1. Objectives: To develop ultraslim product candidates with 17mm circumference with reduced visible sidestream that demonstrate product advantages over Capri and Capri Menthol.
2. Status: The Mark 9 at Bryce-Jewett has been operating using a dual paper feed system. This system was shipped to the Louisville Factory along with the first packer the week of April 24.

The Trim I Model 2 for POL 7185 (Trim V vs. Trim I Model 2 regular) remade due to packaging related defects. The remake of Trim I is in analytical. POL 9089 (Trim V Menthol vs. Trim I Model 2 Menthol) shipped April 25.

POL 7186 (Capri vs. Trim V) is in analytical. POL 9090 (Capri vs. Trim VI - Trim V with increased menthol) is awaiting menthol aging.

Trim V cigarettes are being tested on the MC panel for subjective comparison versus a modified blend designed to reduce the ammonia delivery in smoke.

A request was received for an HTI test of identified Capri and Trim V for the Regulars and identified Capri Menthol and Trim VI for the Menthols. The Trims will be in the Virginia Slims Superslims ad/pack packaging.

3. Plans:

POL/HTI testing
Test Market

On-going
Sept. 1989

B. LOTUS:

1. Objectives: To develop a 24.0 circumference 100mm low sidestream product with 70% reduction in visibility to compete against Vantage Excel.
2. Status: MC Panel testing of the 2I blend with CA and charcoal/CA filters showed no significant differences in liking but the charcoal/CA was rated higher in off-taste.

Higher density (40 cpi 2I blend) cigarettes were made with the standard KC low sidestream paper and increased CMC and MAP paper. These will be evaluated for visibility, subjectives and analyticals.

Three blends have been evaluated subjectively, the 2I blend is more acceptable.

Three casings have been evaluated on the 2I blend with casing 8387-93A (containing Lactic acid) preferred. New aftercuts have been evaluated but the control model with 8544-49 aftercut was preferred.

3. Plans:

Prototype Production/Evaluation	On-going
Consumer Testing	2nd Qtr.'89
Test Market	TBD

IV. PROJECT VALUE ENTRY:

A. Objective: To develop products to compete in the Value Entry Categories

B. Status:

Target II

Direction of project changed from Winston look-alike to Camel look-alike. Famous 2A and 2B blends will be used to simulate Camel in both lights and full flavor configurations..

C. Plans:

Target (Bucks) Ad/Pack Testing	June
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V. CARTIER:

A. Objectives: To develop a Cartier similar to Europe's product for the U.S. market.

B. Status: Currently investigating the Famous blend with three different aftercuts, Virginia Slims Lights filler and B&H Ultra Lights filler at 9 mg tar and the B&H Ultra Lights filler at 6 mg tar. Cigarettes will be made the first week in May.

C. Plans:

Prototype Production/Evaluation	On-going
Consumer Testing	TBD
Test Market	Sept., 1989

VI. PARLIMENT LIGHTS L.S. FTB MENTHOL:

A. Objectives: To develop a Parliment Lights Long Size Flip Top Box Menthol product acceptable to the U.S. Market.

B. Status: Currently awaiting N.Y. approval to ship Ad/pack.
Twenty-day spotting evaluation in progress.

C. Plans:

Ad/pack	2nd Qtr.'89
Test Market	TBD

VII. PROJECT TOMORROW:

A. Objective: Develop improved product candidates utilizing materials, designs and technologies currently under development.

B. Status: Two POL's are planned using Blend N Phase I versus Marlboro KS, both with 27mm tipping. Several tests are planned for internal MC testing.

C. Plans:

Prototype evaluations	On-going
POL testing	2nd Qtr.'89

VIII. B&H 83MM FTB ALL NATURAL:

A. Objective: To develop a B&H FTB in both a 12 and 8mg tar level with 24.8mm circumference, in both regular and menthol using an All Natural blend.

B. Status: Factory trial completed 4/16/89 at MC. Analytical results are favorable. Specifications being generated and will be ready for review the week of May 1.

C. Plans:

Test Market	2nd Qtr.'89
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IX. PROJECT AMBROSIA:

A. Objective: Develop a 23.0mm circumference aromatic sidestream product.

B. Status: Flavor release compounds from Chemical Research and a commercial source are being coated on cigarette paper and incorporated into the sideseam adhesive. Models will be made with the above as well as a pre-coated Ecusta paper using Virginia Slims Lights construction.

C. Plans:

Prototype evaluations	On-going
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PROJECT NUMBER: 4022
PROJECT TITLE: International Product Development - Asia/Pacific Region
PERIOD COVERED: April, 1989
WRITTEN BY: C. B. Altizer

I. PROJECT TANG (Indonesia)

- A. Objective: Develop a subjectively and analytically identical product to convert locally manufactured MF LS from cut filler to BBS without expanded components.
- B. Results: The Richmond Panel has selected an acceptable prototype and sent it to Hong Kong.
- C. Plans: Await Hong Kong management approval. C. Goodwin will discuss necessary modifications to Malang primary with R. Kennedy next month during a visit to Asia.

II. PROJECT TOLSTOY (Various Countries)

- A. Objective: Develop an upscale Russian-style cigarette.
- B. Results: A prototype has been sent to Hong Kong and Taiwan for monadic PMI concept testing.
- C. Plans: Await results of PMI testing.

III. PROJECT CEDAR (Pan-Asia)

- A. Objective: Develop a free-standing cigarette which would capitalize on the growth of the menthol segment as well as compete favorably with Salem Lights.
- B. Results: Prototype development consists of two blends, two flavor systems used in conjunction with mentholated foil in a recessed and flushed filter design.
- C. Plans: Prepare prototypes for possible PMI testing vs. Salem Lights.

PROJECT NUMBER: 4024
PROJECT TITLE: Japan Product Development
PROJECT LEADER: S. B. Nelson
PERIOD COVERED: April, 1989

I. MERIT LIGHTS

- A. Objective: To develop a 7 mg tar product to compete with Mild Seven Lights among mainstream Japanese smokers.
- B. Status: Specifications for Merit Lights KS Box were transferred to Manufacturing. Production of this new packing is planned to begin May 8th with a national introduction in August 1989.

II. LARK 1989 PROGRAM

- A. Objective: To optimize the subjective acceptance of the Lark family while retaining the Lark character.
- B. Status: The Danchi test results of the Lark Milds blend/tar evaluations were received and analyzed. The results indicate that there is no statistically significant difference between the two blends on the liking scale. However, there was statistical difference on the irritation scale with the GLC-3 blend being rated as less irritating. Regardless of blend, Lark Milds at 9.5 mg FTC tar was rated statistically less irritating and less strong than the current Lark Milds at 11.5 mg FTC tar. Both the 11.5 mg and 9.5 mg FTC Lark Milds were equally rated on the liking scale.

Topline results from Lark Milds PMI blend test indicates there is no significant difference between the current blend and the GLC-3 blend among Lark Milds, Cabin Milds and Mild Seven Lights smokers.

The Lark Danchi test to evaluate the current blend versus the GLC-3 blend at lower tar deliveries was shipped. Results are expected the week of June 5th.

The production of the new Lark KS Box packing at 15.5 mg FTC tar was initiated. This product will be introduced in June 1989. Initial shipments of the Lark KS SP and Lark 100's SP with the FTC tar reduced to 15.5 mg/cigt. will arrive in May 1989.

Studies continued to evaluate how to reduce the current level of ventilation variability in the Lark products. Filters with alternative combining wraps and other experimental models were manufactured for evaluation on Lark Milds. These alternatives included Kimberly Clark's porous combining wrap without heat seal. In addition, discussions were held with Dexter to determine if they were able to provide an improved combining wrap for the Lark family. Samples of their commercially available porous wraps with heat seal were presented for our evaluation.

III. BACKGROUND TESTING

- A. Objective: To assess the impact of a recessed filter on Japanese consumers' subjective response.
- B. Status: Results of the Danchi test to study the impact of selected filter designs were received and analyzed. The analysis of the liking scale indicates that the panelists equally rated the standard dual filter, the flush filter with mouthpiece paper and the recessed filter model, when all the models had the same tar delivery. However, the recessed filter model at reduced tar (9.5 mg/cigt.) was rated significantly lower on the liking scale.

IV. CHESTERFIELD

- A. Objective: To develop an 11 to 12 mg tar American blended product.
- B. Status: The repeat PMI test of Chesterfield with GLC-3 blend versus Lucky Strike was shipped. Results are expected the week of June 5th. In addition, cigarettes for a Danchi test to evaluate alternative blends for Chesterfield were made in Semiworks. The four models to be tested are as follows:

- o GLC-3 Blend/Famous B Flavor System
- o Famous Blend/Famous B Flavor System
- o Target Blend/Famous B Flavor System
- o Lucky Strike

Results are expected in June 1989.

V. ULTRA LIGHTS

- A. Objective: To develop an ultra light product for the Japanese market.
- B. Status: Cigarettes were made for a Danchi panel evaluation of two ultra light models. The two models - Mount blend and Natural Blend "E" at 5.0 mg FTC tar/cigt. - will be tested with Caster and Caster Milds.

PROJECT NUMBER: 5001
PROJECT TITLE: Packaging Studies
PROJECT LEADER: H. R. Dunaway
PERIOD COVERED: April, 1989

PACKAGING STUDIES

A. **Objective:** Provide technical packaging support to Manufacturing, Manufacturing Services, Engineering, Purchasing, and Quality Assurance. In addition, assist New Products Directorate in evaluating new packaging concepts and products.

B. **Status/Plans:**

Residual Printing Solvents in Packaging Materials: Coordinated evaluation of the following items:

- Materials sampled at JTI's Odawara factory at various time intervals after printing:

Marl 100 SP 20	3/13, 3/17 & 3/31
Marl KS SP 20	3/9, 3/16 & 3/30
Marl KS FT 20	3/7, 3/14 & 3/28
Marl 100 SP 20/200	3/13, 3/17 & 3/31
Marl KS SP 20/200	3/9, 3/16 & 3/30
Marl KS FT 20/200	3/7, 3/14 & 3/28

- Plus 120 FT 20 & 20/200
- Marl UL 100 Red FT 6
- Marl UL 100 Blue FT 6
- Marl UL KS Blue FT 6
- Marl UL KS Red FT 6
- EEC "Marlboro" Carton Insert
- L&M 100 Men SP 20/200
- Marl Men 100 FT 20
- AOCO 34688 Oasis Over Lacquer (solvent free)
- Nacan Pressure Sensitive Tear Tape
- .012 King James Gemcote Board
- Marl Lts KS SP 20/200 from JTI

Project Art: Models at 8 & 11 mg tar were conditioned to OV levels of 11, 11.5, 12, 12.5, 13, and 13.5% for Flavor Development subjective evaluations.

Fiber-Lam, Inc. Shipping Cases: Plans for a Domestic shipping test have been outlined. Plans for an Export shipping test, and additional controlled testing are under development.

PROJECT NUMBER: 0400
PROJECT TITLE: Low Density Rod Development
PROJECT LEADER: R. S. Mullins
PERIOD COVERED: April 1989

I. LOW DENSITY ROD

- A. Objective: Develop a continuous process for the production of reduced density cigarettes.
- B. Results: Production of Half-Pint type low delivery models was completed for a test comparing the subjective and analytical properties of low delivery cigarettes produced using the low density process to that of cigarettes produced conventionally using high DIET blends. Production of samples to support binder application studies by Project 1503 continued.

Development Engineering completed the design of modified steaming garnitures needed to begin investigations of spray application of binders on the maker. Fabrication of the parts by the machine shop is pending.

Fabrication and installation of a system to apply moisture to the tobacco bed on the suction tape just downstream of the ecreteur was completed.

- C. Plans: Produce low density samples for low sidestream investigations. Produce samples as requested by Project 1503 for binder application studies. Evaluate the impact of moisture addition to the tobacco bed on cigarette quality. Begin investigations into the feasibility of spraying binder onto the tobacco bed on the maker.

II. Kaymich Menthol Application at Maker

- A. Objective: Evaluate the Kaymich menthol applicator as a means of applying menthol directly to either cigarette paper or filler at the cigarette maker.
- B. Results: As reported last month, cigarettes which had molten menthol applied to the cigarette paper lost a significant amount of the menthol upon exposure to air. The menthol content of cigarettes laid on a table in a single layer dropped linearly with time from 4.4 mg/cig immediately after production to 2.6 mg after 3 hours. After reviewing these results with representatives from Operations Services and Quality Assurance, this loss was deemed to be severe enough to prevent effective implementation of the process in manufacturing. Accordingly, no further development of this approach is planned.

Preliminary trials to determine the feasibility of applying menthol to the tobacco bed on the suction tape of the maker were conducted on April 20. No significant maker problems attributable to the menthol application were seen with the air

atomization nozzle. The liquid application nozzle, however, appeared to produce increased choke-ups on the suction tape. Both application modes resulted in severe initial spotting of the cigarette paper from the molten menthol. This initial spotting disappeared relatively quickly as the menthol dried. However, the molten menthol on the paper appeared to wick a brown or yellow substance out of the tobacco which was left on the paper after the menthol dried. There is some indication that these secondary spots also disappear with age. Samples from the trials have been submitted to CTSD for analytical testing and to QA for a spotting evaluation.

- C. Plans: Produce additional samples for product evaluation and for investigations of volatile menthol losses and cigarette spotting.

III. Kaymich Menthol Application to Foil

- A. Objective: Evaluate the Kaymich menthol applicator as a means of applying menthol to foil without the ethanol carrier.
- B. Results: Results from the tests to determine the effects of application temperature on the absorption of menthol by the paper backing and the transfer of menthol to the foil face were encouraging. As the application temperature was increased from 50° C to 90° C the transfer of menthol to the foil face was decreased from 18% to 8% and a corresponding increase was seen in the level of menthol retained in the paper backing.

Sample bobbins have been coated using this technique for an aging test to compare the analytical and subjective properties of mentholated cigarettes produced via this process to those produced with the standard menthol-on-foil process. Packing of sample cigarettes is scheduled for the second week in May.

- C. Plans: Produce product samples for tests to determine the rate of migration of menthol from the foil and to evaluate the subjective impact of this mentholation technique. Install the Kaymich unit on the PM coater being installed in Semiworks. Determine the effect of residence time on foil contamination.

PROJECT NUMBER: 1307
PROJECT TITLE: Reconstituted Tobacco Development
PROJECT LEADER: R. G. Uhl
PERIOD COVERED: April, 1989

I. IMPROVED SHEET PROPERTIES

A. Objective: Improve the physical characteristics and blend properties of reconstituted sheet materials.

B. Results:

1. ART Project - Expert panels have completed screening of the individual sheet materials containing ART stems. Equivalence to controls (individual RL types in 100% and blended cigarettes, and both RL types combined in blends) is indicated with ART stems incorporated as 1.8% of pilot RL feedstock. ART stems are less obvious in the TC flavored sheet, but are still detectable at 3.6% inclusion. Similar evaluations of BL Plant test RCB show that 11% ART stem inclusion is acceptable, but 22% is detectable. Semiworks cigarettes are in preparation to evaluate sheets with the recommended ART stem inclusion levels (1.8% in RL; 11% in RCB) combined in Marlboro blends, plus permutations of this formula, e.g., approximating 16% inclusion in RCB with a mix of the 11% and 22% sheets. The recommended blend would utilize 32% of the ART stems.

ART stems from the bottom absorber basket are subjectively better in RL than top basket stems, implicating the lipids ("wax") layer deposited on the top basket during pressure letdown. The wax-laden top layer of stems (2-3% of the stems) has been accumulated for analysis and will be subjectively evaluated in pilot RL. The non-wax portion (97-98%) of the stems has been made into RL to determine if this material is subjectively equivalent to bottom basket stems. Extensive chemical analysis has shown no measurable differences (other than alkaloids and citrate) between control CRS, monopotassium citrate cased CRS, total ART stems, top basket stems and bottom basket stems. Tandem mass spec analysis might detect chemical differences once this equipment becomes available.

Total ART stems that were thermally treated in the Semiworks Hauni HT tunnel were subjectively preferred over bottom basket stems in pilot RL (compared at 7.2% inclusion in feedstock). These stems have now been included in RL at 3.6% and 1.8% to confirm the benefits of Hauni treatment and to determine if this will allow higher ART stem inclusion levels.

ART stem inclusion in RCB (replacing burley stems) would require increased burley stem usage in RL to maintain durations. Control and ART stem pilot RL sheets will be made in this mode in May for use in blend combinations with ART stem RCB sheets. The ART stems will be Hauni treated if the testing in progress shows this to be subjectively beneficial.

2. Humectants - Preliminary results from subjective evaluation of the four PG/G-free blended cigarette models indicate potential acceptability for PG/G-free strip casings employing reduced isosweet levels (no sucrose); the three casing formulations using other natural flavors still showed differences versus controls. If these results hold, and no additional casing adjustments are necessary, a POL will be initiated using the low level isosweet casings.

C. Plans:

1. Expedite subjective evaluation of blend combinations of RL and RCB materials containing ART stems.
2. Determine if Hauni treatment improves ART stem subjectives and produce pilot RL with feedstocks adjusted to maintain stem durations with ART stem use in RCB.
3. Continue investigating the effects of the ART stem wax layer on sheet subjectives

II. SUBJECTIVE MODIFICATION OF RL

A. Objective: Improve or modify the subjective character of RL.

B. Results:

1. Liquid Flavors - Subjective screening of pilot 150B sheets made with liquid flavors from alternate vendors showed significant differences. The latest sample from Chart was subjectively unacceptable; the prior Chart samples had been consistent, and functioned as the control liquid flavor and the basis for setting specifications. The Takasago sample was acceptable; Takasago presently has only a pilot operation and scaleup to commercial will be required. One of the Madis samples was completely unacceptable (too roasted); the second sample was improved but still requires correction. Flavor Development will continue working with vendors to provide analytical feedback on new samples to be made after modifications to roasting/extraction conditions.

C. Plans:

1. Produce pilot 150B sheets to evaluate new liquid flavor samples once these become available.

III. PAPERMAKING TECHNOLOGY

A. Objective: Develop proprietary cigarette papers for low sidestream and other new product applications.

B. Results:

1. Handsheets - The handsheet lab produced trilayer sheets with filler combinations, bilayer and composite Smellrite (Union Carbide zeolite) papers, high (50%) carbonate sheets, bilayer 50%/0% carbonate papers, and papers filled with magnesium ammonium phosphate, precipitated dolomite, hydrotalcite, or hydrotalcite/calcium carbonate. Most handsheets were then sized with a potassium succinate solution. Handsheets were produced with methylcellulose or sol-gels added as a size or included in the sheet (added to the pulp slurry).

Sol-gels included in the paper slurry (Project Lotus) tend to retard drainage and reduce porosity without affecting paper physical properties. Sol-gels that were filtered, dried and ground showed a marked propensity to agglomerate in storage and when added to the fiber slurry. The first samples of sol-gel provided by New York Polytechnic University performed favorably; there was no obvious agglomeration, sheet formation was good, and the impact on stock drainage rates was much less. This material appears suitable for a pilot paper run at Maine if sidestream performance is acceptable.

2. Pilot Trials - Cigarettes made with Trim-V double-wrap papers, the Maine bilayer paper, and the Maine monolayer composite paper gave comparable sidestream reduction when adjusted for static burn time, indicating that reduction may be a function of the heavy sheet weight (63 gms/m²). In order to clarify this point, comparable monolayer composite papers at the same 63 gm weight are currently being produced at the University of Maine at three calcium carbonate loadings.

Bilayer handsheets (combined after free drainage) had indicated more effective sidestream reduction than composite handsheets. Sheet splitting results from Maine indicate some filler migration between the layers of the Maine papers, implying that bilayers may outperform double-wraps if the filler integrity of the layers can be maintained. This would require two fourdriniers and combining of the layers after free drainage, as opposed to the double headbox arrangement on a single wire as used at Maine.

The Trim-V cigarettes made from the double-wrap, Maine bilayer and Maine monolayer papers were all judged subjectively acceptable, with the bilayer paper preferred ("cleaner"). Marlboro Lights models have also been made with a Kimberly-Clark production paper and a comparable Maine paper in order to subjectively qualify the Maine flax stock and process.

The Maine magnesium carbonate/calcium carbonate bilayer paper was made into cigarettes and is scheduled for sidestream evaluation; subjective screening showed the characteristic magnesium off-taste. Handsheets with 30% hydrotalcite filler gave a black, weak ash; the hydrotalcite papers scheduled for Maine production were modified to a mixed filler of 15%

hydrotalcite and 15% calcium carbonate. Maine has committed to installing a vergeure roll on the second press (to provide watermark lines on the Maine papers) and a stock screen on the secondary headbox.

3. Other Support - The 23" rolls of paper coated with sol-gel at Western Michigan were slit into bobbins at Colonial Heights Packaging. The combination of humidification, redrying and tension during the slitting process removed most of the wrinkles and the appearance of the papers was much improved. The sol-gel patterns became glossy; pattern formation appears to be very sharp. Gravure press cells would have to be three times deeper to achieve the Project Tomorrow coating targets, and the ability of the paper to wick sol-gels out of the deeper cells may be limiting. Humidity control and the avoidance of overdrying may help to combat paper puckering during coating, but the high coating target weights and the high water content of sol-gels aggravate this problem. A non-aqueous sol-gel solvent that does not swell the paper fibers or disrupt sheet structure would alleviate this situation.

C. Plans:

1. Continue the preparation of handsheets to evaluate designed fillers and sol-gel preparations
2. Complete the production of heavyweight papers and papers containing hydrotalcite at the University of Maine.
3. Schedule the production of bilayer papers for the end of May at the University of Maine.

PROJECT NUMBER: 1313
PROJECT TITLE: Semiworks Process Development
PROJECT LEADER: K. H. Cho
PERIOD COVERED: April, 1989

A. Objective: Provide engineering and technical support to improve the performance of the Semiworks operation.

B. Results:

Improvement of Alcohol Storage and Flavor Room (Skidmore) - The required tanks, agitators and explosion proof balance have been placed on order.

2nd Aftercut Cylinder (Skidmore) - The 2nd test for subjective evaluation of the new 2nd A/C cylinder was conducted by the M/C smoking panel. The test results showed there is no difference between cigarettes from 1st and 2nd A/C cylinders.

Small Scale Primary (Skidmore) - A verbal approval for the 650 by upper management was obtained.

Small Blending System (Webster) - The contract has been awarded for fabrication of the new feeder and system installation.

Unused Equipment Removal (Banks) - Unused equipment in the preblending area has been removed and the preblend steaming cylinder has been relocated. The rearranged system has been in operation.

Primary Reconfiguration (Cho) - A preliminary equipment arrangement developed by the Central Engineering Department is being reviewed.

Improved Pack Seal (Osmalov) - The new improved pack seal process has been implemented for 85mm Scandia in Stockton St. plant. A test is being conducted by Stockton St. plant to implement the new pack seal process for 100mm AMF-379/Scandia.

Relocation of Laser Perforation (Osmalov) - A laser perforation system has been removed from Module #3 and installed to Module #5 for Lark product development.

Transfer of Trim Making/Packing Equipment (Osmalov) - Shipment of all Trim making/packing equipment has been delayed until another HTI test is completed.

HID Inspection System Training (Osmalov) - Operator training has been completed for Hauni HID inspection system.

C. Plans:

Ring Tipping Adhesive - Continue selection and evaluation of ring tipping adhesives to improve tipping bond and meet cigarette taste.

Improvement of Cork Tipped Products - Test plan is to be developed to improve matching cork tipped products.

PROJECT NUMBER: 1333
PROJECT TITLE: Semiworks Process Control
PROJECT LEADER: D. A. Phan
PERIOD COVERED: April, 1989

- A. Objective: Evaluate and revise the process control and data acquisition system to improve processing performance and production quality.

B. Results:

Unused Equipment Removal (Oliver) - This is an on-going project to remove unused equipment in the Semiworks primary, in collaboration with the Semiworks Process Development group. For the month of April, the Quester feeder at the 6,000 lbs preblend area was removed and sent to the Stemmy. A typical pin feeder will be installed at this location in the near future. The 3,000-lbs preblend cylinder was relocated to the 6,000-lbs line. The conveyors at the preblend area were also rearranged to accommodate production flexibility. Work is underway to remove the oversized Burley silo and replace with the Turkish silo; and rearrange equipment at the P&S dryer and Burley top casing cylinder.

On-line Moisture Meter Evaluation (Oliver) - Initial results indicated that the calibration of the new MM55 moisture meter from Infrared Engineering is dependent on different tobacco types. A meeting was held with Infrared Engineering to discuss the problems. A new version of software and hardware to improve its performance will be sent to us by June 1, 1989.

Vacuum Conditioner Motor Control Center Replacement (Phan) - Plan is underway to replace the Westinghouse motor control center at the vacuum conditioner due to safety reasons. A job order request has been approved. Equipment will be ordered the week of May 8, 1989. Installation is planned to begin in July 1989.

Make/Pack Electrical Engineering Support (Phan) - Purchase orders have been issued for a second continuous instant hot water system for glue cleaning purposes. This system will be installed in May 1989.

P&S Dryer Process and Improvements (Phan) - Work is underway in collaboration with Semiworks Primary personnel to improve the performance of the P&S dryer. A test run has been performed on May 1 to establish base line data.

Primary Moisture Control (Phan) - Work is underway in collaboration with Semiworks Primary personnel to identify problems with controlling moisture and establish plan of activities and guidelines to improve performance.

- C. Plans: - Continue providing electrical plant engineering support to the Semiworks and conduct routine QA functions.

PROJECT NUMBER: 1503
PROJECT TITLE: Modified Smoking Materials
PROJECT LEADER: W. A. Nichols
PERIOD COVERED: April 1989

I. LOW DENSITY ROD - BINDER APPLICATION

A. Objective: Produce precoated filler via the batch or continuous process to support the current product and process development efforts on the low density rod program. Develop a method of pre-applying binders to the tobacco in a manner that can be scaled up to commercial scale.

B. Results: Sample coated fillers were produced for several Product Development tests. Significant differences in tobacco and pectin buildup in the cylinder were observed in coating different blends.

Additional testing was performed at increased throughputs of 70, 80, and 100 lb/hr. Cylinder operating parameters require additional refinement for higher throughputs.

C. Plans: Further experimentation will be done with spraying parameters to minimize the formation of pectin flakes.

II. BINDER TECHNOLOGY

A. Objective: Investigate the mechanism of filler bonding and stiffness produced by coating. Improve commercial feasibility by examining alternate binders and processes.

B. Results: Dry coating of pectin powder on filler continues to require different reactivation conditions than spray coated filler. To examine the effect, different levels of water overspray are being tested.

Two samples were produced using yucca as a surfactant. Yucca was applied separately and in combination with the pectin. At the level tested, the firmness of the low density cigarettes produced from control and surfactant treated filler were similar. Testing will be conducted with increased surfactant addition.

Improvements were made to the steam reactivation chamber to eliminate condensate problems. Reactivation testing of different levels of pectin addition to filler was started.

C. Plans: The difference in reactivation conditions for sprayed versus dry coated filler will be examined. Surfactants will be tested for effectiveness.

PROJECT NUMBER: 1806
PROJECT TITLE: New Tobacco Processes
SECTION LEADER: S. R. Wagoner
PERIOD COVERED: April, 1989

I. PROJECT ART - PILOT PLANT SUPPORT

- A. Objective: To provide processes for converting and casing stem materials for the Bermuda Hundred Pilot Plant.
- B. Results: Batches of CRS were cased with monopotassium citrate and shipped to the Bermuda Hundred Pilot Plant on a daily basis. These batches consisted of the standard Louisville CRS cut at 150 cpi, as well as Louisville material cut at 53 and 90 cpi. In addition, D Pilot Plant produced CRS (150 cpi) from blends of Louisville stem feedstock and VST-1 grade stems at ratios of 60:40 and 80:20 for use at the Pilot Plant.
- C. Plans: Continue to produce stem products as required by Project ART.

II. PROJECT ART - COMMERCIAL PROCESS DEVELOPMENT

- A. Objective: To conduct trials providing information for development of the ART commercial process.
- B. Results: Project personnel continued to assist Bermuda Hundred with equipment commissioning in the filler and stem processing areas. Both filler and stems were cased with the appropriate solutions in the commercial facility for use at the Pilot Plant. Also, Bermuda Hundred observed a temperature increase in the AB solution after preparation. This was confirmed through laboratory experimentation and calculations based on the enthalpies of the reactants to be caused by the resolubilization of the CO₂ from the headspace into the solution.

Tests were repeated in the MC DIET facility to check for potential nicotine contamination of extracted filler. As before, no increase in nicotine was produced by placing a small amount of extracted filler in an impregnator and running a normal cycle. The conveyor exit the Vibrabin was also "swabbed" as before. (Previously, this had shown a nicotine increase from 0.10 to 0.15%.) This test was conducted after the conveyor had been wiped clean, and no nicotine pickup was noted.

Installation of the Infrared Engineering on-line nicotine/OV monitor in the Semiworks has been delayed due to a faulty keypad on the unit. A replacement is scheduled to be delivered the week of 5/1.

- C. Plans: Assist in the coordination of the Bermuda Hundred start-up with respect to product qualification.

Evaluate the Infrared Engineering monitor (nicotine, moisture) after installation in a Pilot Plant situation.

III. BINDER DEVELOPMENT

- A. Objective: Develop methods to produce binder systems for the foam bonded ends and low density rod programs.
- B. Results: A NaCMC (7LFPH) of slightly higher molecular weight was profiled in the laboratory for foam collapse and solution viscosity. It appears from the lab data that this material may be useable; however, it is at the upper end of acceptable molecular weight. This material costs \$0.50/lb less than the NaCMC 7L2P that has been qualified in the foam bonded ends process. Also, additional lab work was done on dextrans with molecular weights of 15-20K and 50K.
- C. Plans: Continue to provide support as required to the above programs.

IV. HAUNI HT TUNNEL EVALUATION

- A. Objective: Determine the effect of steam conditioning tobacco materials in the Hauni HT steam tunnel prior to drying.
- B. Results: In the spot reduction program, the following critical spots/2000 cig values have been determined by the QE SOLVIC device with five days aging:

	<u>Test 1</u>	<u>Test 2</u>
DIET feedstock		
Control	45.2	32.4
Hauni treated	28.3	7.3
DIET Product		
Control	7.8	15.1
Hauni treated	3.3	1.1
Menthol on filler		
Control	9.0	18.4
Hauni treated	13.2	10.5
Menthol on foil ,		
Control	11.2	39.5
Hauni treated	12.2	37.2

Thus, the DIET materials showed significant reductions in spots compared to the untreated controls, while the results from the menthol runs are not as conclusive.

A memo outlining the test plans for evaluation of the Hauni tunnel as a means of obtaining cigarette firmness improvements was issued. The corresponding Semiworks requests were submitted.

- C. Plans: Conduct 30 day spot counts on all menthol samples.

Conduct a third set of tests processing DIET feedstock and product through the HT tunnel for physical and subjective testing.

IV. TSCI-ASTA SHEET

- A. Objective: To develop a subjectively and physically acceptable reconstituted tobacco sheet using the TSCI process and PM-RCB technology for international application.
- B. Results: Laboratory studies demonstrated that the removal of air entrained in ASTA slurry significantly improved sheet quality by eliminating pinholes in the sheet. Sheet quality was also seen to improve by removal of the calcium phosphate film on the casting surface and the application of a release agent, both of which improved doctoring from the casting surface.

A means of measuring survivability of reconstituted tobacco in the laboratory was devised which is giving encouraging preliminary results. This could be used as a rapid screening device for samples before running the selected samples through the Semiworks to determine survivability. The laboratory method selected the same ASTA samples for parity with RCB as were selected by Semiworks processing.

Work is in progress to measure the rate of pectin release from different tobacco blends and their components of different particle sizes and also their bulk densities. This work was initiated in response to TSCI concerns about the effect of these variables on materials handling and process operation.

- C. Plans:

Establish the reproducibility of the laboratory impact tester to measure filler survivability.

Determine an optimum release agent formulation to improve sheet doctoring from the casting belt for improved filler survivability.

Complete the determination of the bulk density of different blends and particle sizes on material handling characteristics.

Complete the determination of the rate of pectin release from different blends and particle sizes.

Complete the subjective analysis of ASTA products.

Determine the reason for the analytical disparity of TSA and PM which exists for propylene glycol and glycerin in ASTA product.

Support TSA in any future ASTA trials.

PROJECT NUMBER: 1810
PROJECT TITLE: Project ART
PROJECT LEADER: Ravi Prasad
PERIOD COVERED: April, 1989

I. PROJECT ART

- A. Objective: To support commercial plant design and Flavor Development objectives at the Bermuda Hundred Pilot Plant.
- B. Results: Pilot plant runs continued in support of consumer testing. Extractions continue to meet the 97 percent nicotine removal target.

Commercial plant filler was qualified by achieving target extraction level with DL blend filler cut and cased at the Bermuda Hundred Processing Facility. The subjectives were acceptable. Stems cut at Louisville and cased at the commercial plant were successfully used to extract DL blend tobacco within process specifications.

A special test was completed to allow storage study of extracted filler at various temperature, humidity conditions. Three separate stem absorption tests were completed using different stem types/blends, with acceptable absorption efficiencies and pressure drop performance.

Parameters for water column design at steady state conditions were established at the University of Texas and at General Foods Technical Research. The progress is satisfactory and on schedule.

Evaluation of Hinds on-line monitor continued. The data is being generated to allow correlation of nicotine concentration in CO₂ with the filler extraction performance. Data from the pilot plant monitor is being sent to Hinds in order to determine the UV absorption coefficients during the process cycle. These coefficients will then be used to optimize the design for the commercial units. The current plan is for the commercial plant to have dedicated analyzers on the extractor and absorber.

Ultrasonic inspection of the pipe welds has been completed and a majority of weld defects have been repaired. A few minor weld defects will be repaired in May 1989.

C. Plans:

1. Assist in the startup of the commercial plant. The pilot plant will be shut down from May 1 to June 15, 1989, to allow the R&D personnel to assist in the commercial plant startup.
2. Repair all remaining weld defects.
3. Carry out scheduled maintenance and safety inspections.

PROJECT NUMBER: 1101
PROJECT TITLE: Entomological Research
PROJECT LEADER: D. L. Faustini
WRITTEN BY: L. Ryan
PERIOD COVERED: April, 1989

I. METHOPRENE

- A. **Objective:** To determine the most efficient use of the insect growth regulator, methoprene within PM's cigarette beetle (CB) management program.
- B. **Results:** Research objectives of interest to PM, concerning methoprene (Kabat® and Dianex®) were discussed with Zoecon® representatives (1). The examination of stems treated with Kabat® at 1-10 ppm range and stored for 3 months revealed 100% CB control for all treatment groups (2).
- C. **Plans:** Issue an interim memo to update results on the stem protection study; investigate a quantitative bioassay; and evaluate potential methoprene levels in an M/C environment (using D-Pilot as a model) following a directed cold aerosol application of Dianex®.
- D. **References:**
1. Faustini, D. L. Visit by Zoecon® Personnel - March 28, 1989. Memo to Distribution. March 10, 1989.
 2. Lehman, R. M. Notebook No. 8740, p. 24-26.

II. ECOLOGY

- A. **Objective:** Relate cigarette beetle ecology to tobacco processing and CB control.
- B. **Results:** Discussions regarding new stored product insect pheromones and traps were held with a representative of Trécé.

Focusing the PM USA Integrated CB control program is underway (1) fueled by data gathered during the 1988/1989 global ecology study (2).

An analyses and summary of insect-related consumer complaints was prepared supporting an earlier study. Results showed that the predominance of insect damage to finished goods occurs in the coastal south of the U.S. and in cigarettes that are in the normal retail distribution channels for longer than 6 months (3).

To determine trap efficiency and relate relative to absolute population density requires marked CBs. The marked CBs to be used are black body color mutants of the CB. Black body CBs are not

trapped as well as normal CBs probably due to differences in tarsal setae (4).

- C. Plans: Continue the global monitoring study; determine trap efficiency; and relate trap CB captures to actual CB population density.

D. References:

1. Ryan, L. and Faustini, D. L. The Integrated Pest Management Program at PM USA. Memo to McCuen, R. W. April 6, 1989.
2. Ryan, L. Global Ecology Program-Update. Memo to Faustini, D. L. December 8, 1988.
3. Ryan, L. Insect-related Consumer Complaints (CC) - 1988. Memo to Spielberg, C. T. March 16, 1989.
4. Minor, M. F. Notebook No. 8539, p. 75.

III. FUMIGATION

- A. Objective: Investigate the use of controlled atmospheres (CA) to fumigate tobacco.
- B. Results: 100% control of T. confusum and Ephestia sp. was achieved during the second CO₂ fumigation field trials at Miller Brewings', Fort Worth facility (1). The CO₂ fumigation parameters were 70% CO₂ for 3 days at ambient temperatures inside the silo of 102 ± 8°F (40°C).

Laboratory studies of simulated CO₂ fumigations in bulk storage warehouses were reported (2) and concluded that 60% CO₂ for 8 days at 80°F (27°C) and 65% RH kills 100% of all exposed CB life stages.

- C. Plans: Continue to integrate CA fumigation with PM's CB control program. Present to management a recommendation regarding the use of CO₂ to fumigate bulk storage warehouses.

D. References:

1. Faustini, D. L. Mortality of Ephestia sp. and Tribolium confusum during second CO₂ fumigations. Letter to Logan, R. & High, M. March 17, 1979.
2. Ryan, L. The Application of Carbon Dioxide to Control Cigarette Beetles In Bulk Storage Warehouses - Laboratory Trials in Relation to Field Data. Special Report# 89-020.

IV. SERVICE TO OTHERS

- A. Objective: To provide technical services to areas outside R&D.

- B. Results: Arrangements are in-hand to continue the training of PME's sanitarian (1-3).

A moisturizing cylinder at Stockton Street was evaluated for CB kill as part of the Bulk Tobacco Handling program (4).

CBs were supplied to QA at Cabarrus and Richmond for routine efficacy tests.

- C. Plans: Continue to provide services as requested and approved by R&D management.

D. References:

1. Faustini, D. L. Training of Jean-Marc Freymond. Memo to Lopes, F. April 5, 1989.
2. Ryan, L. The Presence of Jean-Marc Freymond in PM USA Manufacturing and Processing Facilities - April, 1989. Memo to Distribution. April 13, 1989.
3. Ryan, L. J.-M. Freymond's Itinerary - April, 1989. Memo to Faustini, D. L. April 10, 1989.
4. Ryan, L. & Lehman, R. Cigarette Beetle Mortality in a Moisturizing Cylinder at Stockton Street. Memo to Rowe, N. R. March 8, 1989.

PROJECT NUMBER: 1620
PROJECT TITLE: Electrophysiological Studies
PROJECT LEADER: F. P. Gullotta
WRITTEN BY: C. S. Hayes
PERIOD COVERED: April, 1989

I. NASAL EVENT-RELATED POTENTIALS (NERPs)

- A. Objective: To develop methods to objectively and reliably evaluate human responses to smoke constituents and tobacco flavorants.

B. Results:

Cognitive NERP Study

Current experimental efforts are directed toward assisting Flavor Development by utilizing the cognitive NERP paradigm to evaluate various sources of natural menthol and synthetic menthol substitutes for natural menthol. Subjective testing is also being conducted on four menthol samples to determine whether subjects can detect differences. Further NERP and subjective testing needs to be conducted before any conclusions can be derived from the data.

Experiments comparing a 75:25 mixture of synthetic/natural menthol to 100% synthetic menthol continue to be conducted. To date, three subjects have been tested with this comparison.¹ All of the subjects failed to correctly identify the different stimuli, indicating that the comparison was very difficult to discriminate. This more difficult discrimination should allow us to determine the sensitivity of the NERP technique for discriminating differences among flavorants.

- C. Plans: Continue NERP and subjective testing of various synthetic and natural menthol substitutes for Flavor Development as requested. Complete testing of ten subjects in the 75:25 synthetic/natural menthol mixture vs synthetic menthol comparison.

D. Reference:

1. Martin, B. Notebook No. 8689, pp. 160-178.

PROJECT NUMBER: 1702
PROJECT TITLE: Optical Processing and Aerosol Research
PROJECT LEADER: K. A. Cox
PERIOD COVERED: April, 1989

I. PACK INSPECTION

- A. Objective: Develop and implement a method for the on-line inspection of cigarette packs.
- B. Results: Three enhancements to the inspection algorithm have been developed. First, a change has been made to represent the edge-enhanced images in a binary, bipolar format. Second, the image will be divided into a large number of segments (e.g., 256) and a discriminant filter applied to each segment. These two changes are expected to significantly increase the inspection capability of the system, allowing the detection of small blemishes which previously went undetected. Finally, a new centering algorithm has been developed which will allow more precise positioning of the image prior to the application of the discriminant filter. The new centering method is much more tolerant of the nature of the region chosen for carrying out the centering. The method is also expected to be useful for the off-line inspection of package blanks.

Approximately half of the images of Lark packs captured on video tape from a camera on an ITRAN system connected to a continuous conveyor were digitized with the Androx image computer.

- C. Plans: Complete the digitization of the Lark pack images. Evaluate the capability of the inspection algorithm for inspecting the images.

II. CUT CLOSURE STAMP INSPECTION SYSTEM

- A. Objective: Develop and implement a system for the off-line QA inspection of cut closure stamps.
- B. Results: The computer program for the analysis of the cut closure stamp images has been written and debugged. This program allows the Sun microcomputer to control the stepper motor that turns the stamp handling system and capture images from the video camera. Tests indicate that the inspection system can correctly classify cut closure stamps. In one test 1004 stamps were inspected, including 5 stamps that were turned with their backs presented to the camera. All stamps were correctly classified with no misfeeds. Although the test was promising, other tests have shown that the stamp handling mechanism will occasionally feed two stamps at once. A circuit has been designed to detect this occurrence.
- C. Plans: Complete the development of the stamp inspection system and evaluate it in the QA environment.

III. INDIVIDUAL CIGARETTE INSPECTION

- A. Objective: Develop methods for the on-line inspection of individual cigarettes.
- B. Results: The AccuRay CIM was used to obtain characteristic curves for letters on a simulated cigarette rod. The ACRO 916 computer programs were modified to permit data grabbing to be triggered by the sample. The ACRO 916 limited viewing to just three output ports; however, the initial data demonstrate the feasibility of the data acquisition method.
- C. Plans: Obtain characteristic curves for actual cigarette print.

IV. HIGH SPEED PRINT INSPECTION

- A. Objective: Develop methods for the inspection of print on a printing press.
- B. Results: For the inspection of print produced at very high rates, (greater than 200 images/sec), a self learning scheme by which the inspection system can learn adaptively is highly desirable. Such a scheme has the advantage of requiring minimal input from an operator, as well as being able to adjust for any gradual, acceptable changes in the image. The Widrow-Hoff adaptive learning algorithm has been successfully applied to the generation of a discriminant filter for the inspection of images of the Marlboro pack. The filter generated was found to be nearly identical to the filter generated earlier by our batch training procedure.

PROJECT NUMBER: 1704
PROJECT TITLE: Supercritical Fluid Processes
PROJECT LEADER: T. M. Howell
PERIOD COVERED: April, 1989

I. LOW NICOTINE

- A. Objective: Develop second generation processing for ART.
- B. Results: The eighteen extraction runs to produce enough filler for machine made cigarettes have been completed. One pound of extracted filler was produced using stems as absorber, one pound using carbon as absorber and one pound using water as absorber. Nicotine removal was 95%, 96% and 97% respectively using standard conditions of 3% AB and 200 M/M. Preliminary subjective evaluation indicates parity between stem extracted and water extracted while the carbon extracted was noticeably different. The final report is being prepared. Chemical analyses of the filler and absorbers are pending.

Improvements to the laboratory water absorber unit, specifically the addition of a view cell as a de-misting volume which allowed for increased flow rate, resulted in obtaining 97% extraction of nicotine at 150 M/M using 3% AB. Reduction of AB addition to 2.2% gave 96% nicotine extraction at 200 M/M. Future runs will be made in order to evaluate the effects of other process parameters on the extraction using water as absorber.

- C. Plans: Work is on going.

II. LOW NICOTINE

- A. Objective: Support to ART commercial plant.
- B. Results: It was previously reported that the presence of oxygen in water containing chlorides enhanced the probability of stress corrosion cracking in 316 stainless steel. The chances were reduced if the level of oxygen was reduced to 0.01 ppm or lower in the water phase. Since any free water present in the system is likely to contain tobacco solubles a plan was developed to determine how they affected the distribution of oxygen between carbon dioxide and water. Samples from the BHPP have been taken and water extracts of filler and stems were obtained to be used as the test liquor. An oxygen analyzer was received and is currently being calibrated.
- C. Plans: Work is on-going.

PROJECT NUMBER: 1708
PROJECT TITLE: Physical Chemistry and Process Monitoring
PROJECT LEADER: J. L. Banyasz
PERIOD COVERED: April, 1989

I. OPERATIONS SUPPORT (J. Crump and A. Closter, in collaboration with the Applied Technology Group)

- A. Objective: Determine the effects of particle size distribution on dynamic viscosity changes that occur in PVA tipping adhesives.
- B. Results: The Mark 10A simulator is currently being set up and preparations made to begin testing. Testing of the effect of roller hardness on the viscoelastic properties of tipping adhesives has been initiated with the Mark 9 simulator. Rollers have been fabricated from rubber of varying hardnesses (Durometer ratings of 20, 50, and 95). The preliminary results indicate that roller hardness has an effect on the rate of viscosity change during machining.

II. MENTHOL STUDIES (T. V. Van Anken)

- A. Objective: Determine the diffusion rate and solubility of menthol in cellulose acetate.
- B. Results: The facilitation by water of menthol sorption in CA has been confirmed. The effect appears to be a kinetic phenomenon.

III. OPERATIONS SUPPORT (P. Henderson, in collaboration with the Applied Technology Group)

- A. Objective: Characterization of inks.
- B. Results: The three month factory "operating window" study has been completed. The data is being collated and evaluated to determine the direction of future work.

IV. OPERATIONS SUPPORT (D. Driscoll in collaboration with the Applied Technology Group)

- A. Objective: Characterization of side seam adhesive application.
- B. Results: A simulator for side seam application with flow measurement and control has been installed.

The initial work to define flow as a function of reservoir size, reservoir configuration, and nozzle size has been completed. The data is currently being assessed to determine future work.

PROJECT NUMBER: 1720
PROJECT TITLE: Analytical Microscopy
PROJECT LEADER: V. L. Baliga
PERIOD COVERED: April, 1989

I. LOW SIDESTREAM CIGARETTE PAPERS (BALIGA, SANDERS, HENRY)

- A. Objective: Examine the ultrastructure of selected cigarette papers and paper additives in support of the low sidestream project.

B. Results:

Sol Gel Coated Paper and Ash: Sol gel that was applied to the paper using a size press formed a continuous coating of equant-shaped particles on the flax fibers and the CaCO_3 filler. They were encased within the sol gel coating. The unit particle size of the sol gel was $0.02\mu\text{m}$ to $0.11\mu\text{m}$ in diameter. The sol gel was found on both sides of the paper.

Ash: The ash of the sol gel coated paper appeared as an interconnected network. Its unit structure was similar to that of the sol gel on unburned paper.¹

Precipitated Sol Gel: Two samples of Al sol gel and one sample of Ca-Al sol gel were examined for shape, size, and elemental content. All three sol gels were precipitated, dried, and ground. Aluminium sol gel, #8741-82-1P, contained particles of differing sizes no greater than $45\mu\text{m}$. The large particles consisted of unit particles which measured $0.01\mu\text{m}$ to $0.15\mu\text{m}$ in diameter. Aluminium was the only element found. Sample #8741-78-1 consisted of particles of differing sizes and shapes, but the majority of the large particles were angular in form and were no larger than $30\mu\text{m}$ in length. These large particles also consisted of equant-shaped unit particles that measured $0.02\mu\text{m}$ to $0.11\mu\text{m}$. Aluminium was the only element found. The Ca-Al sol gel was distinctly different from the Al sol gels. The gross structure consisted of rounded particles which measured $0.2\mu\text{m}$ to $2.0\mu\text{m}$ in diameter. This round form was the result of many loosely packed, platelet-shaped discs that measured $0.02\mu\text{m}$ to $0.04\mu\text{m}$ thick. More Al than Ca was detected in this sample².

Chemical Additives: Three chemical additives were examined to determine size, shape and elemental composition. They included 1. $\text{Ca}_{10}(\text{OH})(\text{PO}_4)_6$, 2. $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$, and 3. CaHPO_4 . Sample 1 contained equant-shaped particles $0.02\mu\text{m}$ to $0.15\mu\text{m}$ in diameter which formed larger clumps no greater than $3.0\mu\text{m}$ in diameter. Sample 2 contained two types of crystal shapes, one an equant-shaped particle, $0.05\mu\text{m}$ to $0.17\mu\text{m}$ in diameter while the other was angular and columnar in shape which measured $0.17\mu\text{m}$ to $50\mu\text{m}$ in length by $0.07\mu\text{m}$ to $21\mu\text{m}$ in width. Sample 3 contained very angular rhombohedral structures which ranged in size from 1 to $37\mu\text{m}$ along the two dimensions while the third dimension was about half that of the other dimensions. All three chemicals contained Ca and P^3 .

Precipitated dolomite was examined and found to consist of equant-shaped particles that measured $0.05\mu\text{m}$ to $0.15\mu\text{m}$ in diameter. A few of the particles contained straight edges but no differences in

elemental content were noted. All particles contained about three times more Ca than Mg^4 .

Aspen Fiber Paper with Precipitated $CaCO_3$: A handsheet, made at the University of Washington, was made from Aspen fibers and $CaCO_3$ filler that was produced by precipitating $Ca(OH)_2$ with $Na_2CO_3^5$. The structure of the precipitated $CaCO_3$ was distinctly different from the standard barrel-shaped $CaCO_3$. This $CaCO_3$ was ovoid in shape and measured about $1\mu m$ by $3\mu m$ similar to the barrel shaped $CaCO_3$. However, the ovoid structures were made up of platelets stacked one on the other. Each ovoid particle contained 50 or more stacked platelets. The $CaCO_3$ particles were closely associated with microfibrils as if the microfibrils were sites of nucleation. The $CaCO_3$ associated with the ash had not changed in structure from the unburned $CaCO_3$. The aspen fibers did not contain any $CaCO_3$ crystals within the lumen of the fibers⁴.

C. References:

1. Baliga, V., "Characterization of Sol Gel Coated Cigarette Paper and Ash," Memo to C. Kroustalis, April 4, 1989.
2. Sanders, K., "Analysis of Sol Gel Samples," Memo to A. Kallianos, April 10, 1989.
3. Sanders, K., "Structural and Elemental Documentation of $Ca_{10}(OH)_2(PO_4)_6$, $CaHPO_4 \cdot 2H_2O$, and $CaHPO_4$," Memo to J. Paine, April 18, 1989.
4. Baliga, V., "Structural and Elemental Determination of Precipitated Dolomite," Memo to S. Baldwin, April 14, 1989.
5. Bokelman, G., "Report on February Trip to the University of Washington," Memo to R. Ferguson, February 28, 1989.
6. Baliga, V., "Characterization of Aspen Fiber Paper with Precipitated $CaCO_3$," Memo to G. Bokelman, March 21, 1989.

A. Objective: Develop procedures to measure visible sidestream smoke and plot visible sidestream versus time.

B. Results: A program written by J. Russ to gather brightness data has been revised so that the data collected is stored as raw data of gray scale values. The data collected at a single point in time is stored as a frame. With the revised program the raw data can be presented as a brightness value for one or more cigarettes. It also can be used to plot the dynamic profile of smoke produced versus time, for one or more cigarettes¹.

C. References:

Henry, B., "Program for Summing Sequence Data for Sidestream Testing," Memo to V. Baliga, March 23, 1989.

II. SAFETY (SANDERS)

A. Objective: Serve as the R&D First Aid Team Captain and member of the Emergency Response Team.

B. Results: Emergency Medical Technician classes are being taught on a weekly basis.

PROJECT NUMBER: 1752
PROJECT TITLE: Optical Spectroscopy of Tobacco and Smoke
PROJECT LEADER: G. Vilcins
PERIOD COVERED: April, 1989

I. SMOKE CHEMISTRY OF REDUCED DENSITY ROD CIGARETTES

- A. Objective: To analyze the smoke condensates from low density rod cigarettes.
- B. Results: The smoke data from the low density rod (LDR) model (X6D9GL) and three controls (X6D9GM, X6D9GN, and X6D9GK) were scaled to a per mg CSC, per puff and per cigarette basis. Factor Analysis in BMDP was used to process the data because of its ability to compare the four cigarettes simultaneously. Different scaling methods yielded the same information. In basic fraction, Factor 1, containing volatile pyridines, pyrazines, quinolines, and quinoxaline as the main contributors, separated the cigarettes with and without pectin. The cigarettes with pectin delivered higher amounts of these bases per mg of condensate. The increased delivery of the volatile bases could be due to the citrus pectin providing an additional source of reducing sugars for temperature-induced browning reactions. Factor analysis of the acidic fraction was dominated by one factor. Factor 1 again differentiated the cigarettes by the added citrus pectin. The increased delivery of several simple phenols in the K and L cigarettes probably was due to the breakdown of the added citrus pectin. However, none of these compounds were found to be significantly different. The differentiation of the cigarettes by the neutral fraction also was driven by the pectin factor. The pectin-containing cigarettes delivered more sugar-related degradation products, such as indole, cyclopentanones, furans, and furfural than the non-citrus pectin cigarettes.
- C. Conclusions: The smoke chemistry in the low density cigarettes mainly is driven by the presence of the additional citrus pectin. By comparing with the controls, the pectin increased the delivery of many volatile bases, simple phenols, and sugar degradation products with the basic compounds being the most influential. The simple phenols and sugar degradation products result directly from the breakdown of pectin while the basic substances probably are due to the browning reaction from the additional reducing sugar source. Even though the added pectin evidently was involved either directly or indirectly in the smoke chemistry, the differences it caused were very small.
- D. References:
- Hsu, F. and A. Oti, "Smoke Chemistry of Reduced Density Rod Cigarettes," memo to R. Kinser, March 8, 1989.

II. ANALYSIS OF CARBON/IRON SAMPLES BY MOSSBAUER AND LASER MICROPROBE MASS SPECTROMETRY

- A. Objective: To evaluate the spectra obtained from the laser microprobe mass spectrometry (LAMMA) analysis.
- B. Results: The LAMMA spectra were obtained at low and high laser energy to identify elemental and molecular composition, respectively. The microscopic differences in elemental composition of a sample were measured at a spatial resolution of approximately 5 μm .
- C. Conclusions: The LAMMA analysis was more useful in characterizing the elemental composition than the molecular composition. The capability of LAMMA to probe the microscopic differences in a sample complements the structural information obtained by Mossbauer spectroscopy and x-ray diffraction spectroscopy. A possible matrix effect prevented a more detailed interpretation of most of the spectra.
- D. Plans: Group discussion of LAMMA results.
- E. References:

Shafer K., "Analysis of Heat Source Materials by Laser Microprobe Mass Spectrometry," memo to C. Lilly, April 11, 1989.

III. ANALYSIS OF OXYGEN IN ART SAMPLES

- A. Objective: To determine the partition coefficient of oxygen in the aqueous and gaseous phase of ART pilot plant drain water under different experimental conditions.
- B. Results: Instrumentation was set up for the electrochemical detection of oxygen. Oxygen concentrations were measured in the aqueous and gaseous phase after bubbling air and carbon dioxide through the water, respectively.
- C. Conclusions: Electrochemical detection of oxygen in both phases is possible using the Clark electrode.
- D. Plans: The electrochemical approach will continue.
- E. References:

1. Howell, T., "Oxygen Solubility in Pure Water Versus ART Pilot Plant Drain Water Containing Tobacco Solubles," memo to E. B. Fischer, April 4, 1989.
2. Shafer, K., PM Notebook #8789, p. 9.

IV. THERMOGRAVIMETRIC ANALYSIS OF GLUCOVANILLIN AND PROPENYL GUAETHOL GLUCOSIDE

- A. Objective: The objective of this experiment was to find similarities in the thermal decomposition characteristics of the two compounds for the feasibility of substituting glucovanillin for propenyl guaethol glucoside.
- B. Results: The samples were analyzed in both TGA and DSC in nitrogen atmosphere with a flow rate of 5°C/min. The two compounds showed similar decomposition patterns both showing evidence of a two step decomposition which is probably the cleavage of the compounds to form vanillin. The only major difference between the two compounds is the amount of residue left after the runs were completed. The residues of the pyrolytic decomposition of propenyl guaethol glucoside and glucovanillin were 1.0 and 10.8 percent, respectively.
- C. Conclusions: The TGA and DSC showed similar decomposition patterns. Further studies should be done by evolved gas analysis to investigate the decomposition products.
- D. References:

Chung, C., "Thermal Analysis of Propenyl Guaethol Glucoside and Glucovanillin," memo to J. Chan, March 22, 1989.

PROJECT NUMBER: 1754
PROJECT TITLE: Spectroscopic Studies of Tobacco and Smoke Components
PROJECT LEADER: J. B. Wooten
WRITTEN BY: R. L. Bassfield
PERIOD COVERED: April, 1989

SOLUTION NMR

- A. Objective: Determine the stereochemistry and structural assignment of three bicyclic lactones (K. Podraza).
- B. Results: ^1H and ^{13}C nmr spectra have been taken on three bicyclic lactones. The stereochemistry at the ring juncture for all three compounds was deduced by means of the ^1H chemical shifts and coupling constants of the two protons at the ring juncture. The structural assignments were made through the use of two dimensional ^1H - ^{13}C correlation (COSY) and ^{13}C - ^1H correlation (HETCOR) experiments.
- A. Objective: Monitor the reaction of glucose and menthyl chloroformate (MCF) in pyridine by ^{13}C NMR (Y. Houminer).
- B. Results: The starting materials and reaction mixture were examined by ^{13}C nmr. ^{13}C spectra were run on the reaction mixture after 1, 3, 5 and 16 hours in the spectrometer. No significant changes were detected in the mixture as a function of time. ^{13}C spectra were collected on the same sample heated to 45°C for 1 hr and again after 16 hours. The only significant difference between the heated sample and the unheated sample was an increase in the peak intensities of the menthol carbon signals.
- C. Conclusions: The reaction of glucose and MCF appears to be complete after the addition of MCF (over 3 hours) as evident by no change in the ^{13}C spectra with time. At 45°C and over an extended period of time, there is degradation of some of the product to form free menthol.

PROJECT NUMBER: 1757
PROJECT TITLE: Analytical Flavor Specifications
PROJECT LEADER: M. L. Zimmermann
PERIOD COVERED: April, 1989

ANALYTICAL FLAVOR SPECIFICATIONS

- A. Objective: To develop analytical and sensory specifications for incoming flavors and materials for use at the Flavor Center and other QA facilities. To provide analytical certification on export flavors manufactured at the Flavor Center for FRG compliance.

B. Results:

The analysis of Direct Materials for compliance to FRG continues to require a large commitment of time. These have involved the numerous changes in the formulation of previous flavor samples as well as the examination of alternative vendors. Vendor retains and preshipment samples are now received on a regular basis and these are given high priority to determine acceptability from a certification standpoint.

A remake of a PMI sample was received and found to be unacceptable. All of the individual components were again requested and analyzed in a joint venture with the Flavor Center. A single component was found to be the source of the contamination. All flavors currently on hand at the Flavor Center will be examined in a joint venture with the Flavor Center doing a preliminary screening process. In an effort to determine the extent of the contamination a series of samples were analyzed by Flavor Development (B. Hale). Vendor samples were again found to be the source.

Samples were received from one vendor in response to the specification program representing each flavor purchased to compare results and define acceptability ranges. Examination of the results remains to be done.

W. Harvey has begun work on the ion chromatograph for one of the marker compounds. Results show that this procedure can generate results consistent with those for GC/NPD and with a lower limit of detection.

- C. Plans: Continue the certification of the PMI samples, begin the certification of all flavors currently on hand at the Flavor Center and continue the specification work for the second vendor.

PROJECT NUMBER: 1758
PROJECT TITLE: Tobacco Cell Wall Research
PROJECT LEADER: G. H. Bokelman
PERIOD COVERED: April, 1989

I. SIDESTREAM REDUCTION (S. Baldwin, S. Tafur, B. Rogers and G. Bokelman)

- A. Objectives: (1) Evaluate machine-made cigarettes containing mono- and bilayer cigarette papers produced at the University of Maine and (2) prepare and evaluate, for reduction in sidestream smoke, paper handsheets that contain coatings and/or inorganic fillers with different physical and chemical properties.
- B. Results: Several papers prepared at the University of Maine were coated with potassium succinate, made into machine-made cigarettes and evaluated subjectively in cooperation with Barbro Goodman. A comparison was made in percent reductions in absorbance (vs. a Marlboro Lights control) for cigarettes made in the Trim V configuration using the standard dual wrap, a bilayer paper (~63 g/m² basis weight, 6.3 Coresta inherent porosity perforated to 30 Coresta) and a mono-layer "composite" paper (~63 g/m² basis weight, 6.8 Coresta inherent porosity perforated to 40 Coresta). The results were as follows: (1) dual wrap, 78% reduction; (2) bilayer, 68% reduction; and (3) mono-layer "composite", 65% reduction. However, preliminary indications from the low sidestream subjectives panel indicated an apparent preference for the bilayer paper. The relatively good performance of the composite paper was surprising. Additional high-basis weight mono-layer cigarette papers will be prepared at the University of Maine during the last week in April.

Thermal studies designed to determine the effects of "fluxing agents" on paper were continued. Thermal analysis (TGA) of all flax papers impregnated with different salts showed several interesting differences in the cellulose decomposition profiles of the various papers (1). Equal molar amounts of potassium were added to the papers as different salts: KHCO₃, potassium succinate and KCl. MgCl₂ and FeCl₃ were also impregnated in other flax sheets. The same flax was used throughout, at a constant porosity and basis weight. The onset temperature for cellulose decomposition was lowered in all cases, as was the temperature of maximum decomposition. Different salts lowered these temperatures by different amounts. Differences in refining were found to have no effect on the thermal decomposition of the cellulose.

BET surface area determinations were performed on numerous samples, including: (1) Mg(OH)₂ (samples from both Morton Thiokol and Reheis), (2) hydrotalcite, (3) Al(OH)₃/Mg(OH)₂ co-dried powder (Reheis), (4) Al(OH)₃ dried from paste (Reheis), (5) four precipitated sol-gel samples from Andy Kallianos, (6) calcium oxalate, and (7) aluminum oxalate. In addition, samples of hydrotalcite to which potassium succinate had been adsorbed (5% and 10%) and water-treated controls were analyzed at room temperature, 200° C and 450° C. This experiment was conducted to determine if the addition of the potassium succinate would "flux"

the filler upon heat treatment and consequently show a decrease in surface area. The surface area of the treated hydrotalcite samples increased with increasing temperature and did not show a "fluxing" effect under the analysis conditions used in this experiment. Further analyses at temperatures $>450^{\circ}\text{C}$ will be conducted.

The BET surface areas of several paper samples also were examined.

A series of twelve cigarette samples were prepared in Marlboro configuration with 2I blended filler. There appeared to be a slight increase in percent sidestream light extinction for the cigarettes with 2I, instead of Marlboro, filler. In another set of experiments, handmade cigarettes with Marlboro Lights filters were found to give a slight decrease in percent sidestream light extinction compared to similar cigarettes with Marlboro filters. From now on all handmade cigarettes will be prepared with 2I blended filler using ring-tipped Marlboro Lights-like 27 mm filters with ~35% dilution.

Aluminum and magnesium contents in three different mineral samples were obtained. For these samples it was found that sidestream reduction improved with higher magnesium content in the mineral.

X-ray fluorescence analysis of both the wire side and felt side of $\text{MgCO}_3/\text{CaCO}_3$ bilayer papers prepared at the University of Maine showed that CaCO_3 was present in both sides of the paper, although the MgCO_3 was located only in the bottom half of the paper. Atomic absorption results for Ca and Mg in the split halves of the paper showed the same result. These distribution results are probably due to the fact that at the U. of Maine bilayer papers are prepared using two headboxes on a single fourdrinier wire. Thus the slurry from the second headbox filters through the first layer. In true commercial production two separate wires would be used, which should minimize intermixing of the two layers.

A statistically designed experiment to be performed with Kimberly-Clark has been initiated (2). The effect of filler surface area, paper porosity, and fluxing agent level on sidestream smoke production will be evaluated by multiple regression analysis.

Additional work on developing a method for coating insoluble inorganic fillers on cigarette paper using the handsheet labsize press was conducted.

A trilayer handsheet was prepared in the handsheet laboratory with two high surface area CaCO_3 fillers as the outer layers and MgCO_3 as the inner layer. This paper will be used to prepare handmade cigarettes for evaluation of sidestream reduction.

Several experimental cigarette papers shown previously to produce low sidestream visibility were re-examined at lower potassium succinate levels. The data indicated generally less sidestream reduction at lower potassium succinate levels for the papers examined. The dolomite papers, however, continued to perform well at the lower levels of potassium succinate.

Analytical results on Kimberly-Clark paper samples intended as standards for QA were obtained from General Analytical. Potassium succinate levels were determined by two independent techniques, GC determination of succinic acid and atomic absorption determination of potassium, both of which correlated well together. Unfortunately, these results did not agree well with the conductivity measurements obtained by Kimberly-Clark. A tentative decision has been made to use the GC procedure as the standard for succinate levels.

New machine-made control cigarettes prepared with 2I blended filler, 27 mm filters with ~35% dilution, and low sidestream paper [35% Mg(OH)₂] or high sidestream paper [137-1] have been received. These cigarettes will provide low and high reference points for sidestream light extinction analyses.

Construction of a diffusional conductance apparatus was requested from the development engineering group. This instrument should be ready in May.

- C. Plans: Additional high-basis weight, mono-layer ("composite") cigarette papers will be prepared at the University of Maine. Handmade cigarettes will continue to be prepared from numerous types of handsheets for evaluation of sidestream reduction. Emphasis will be placed on handsheets with high basis weight, high CaCO₃ content, MgCO₃, and dolomite. Studies will be conducted to further evaluate the effects of water-soluble thermoplastic polymers on sidestream smoke and ash integrity.

D. References:

1. Chung, C., memo to S. Baldwin, "Effects of Metal Ions and Anions on Cellulose Pyrolysis and Combustion," April 7, 1989.
2. Baldwin, S., memo to E. B. Sanders, "A Statistically Designed Experiment to Quantify Paper Parameters Affecting Sidestream Smoke--A Collaborative Research Project with Kimberly-Clark," March 31, 1989.

PROJECT NUMBER: 1902
PROJECT TITLE: Cell/Tissue Culture Research
PROJECT LEADER: I. L. Uydess
WRITTEN BY: M. Shulleeta
PERIOD COVERED: April, 1989

I. ALTERNATE PRESERVATIVE PROGRAM

A. Objective: To develop procedures and to establish microbiological screens for the evaluation of new, nature-identical preservatives as replacements for and/or as adjuncts to propylparaben.

B. Status:

1. Phase I Screens:

Four compounds, 1-carvone, d-carvone, carvacrol and citronellol were tested in a preliminary set of agar inclusion screens to determine the relative antimicrobial activities of each of these nature-identical compounds compared to the standard propylparaben control (@ 500 µg/ml). *B. licheniformis* and *B. coagulans* served as the target organisms. The initial results indicated that while 100 µg/ml carvacrol was sufficient to result in a total inhibition of growth, 500 µg/ml of citronellol were needed to produce the same effect. The two isomers of carvone were not found to be good candidates for further study since they were observed to be ineffective in this assay below 5000 µg/ml.

As a result of these observations, both carvacrol and citronellol were subsequently tested in the Phase I shake-flask assay in order to more accurately assess the efficacy of these compounds against *B. coagulans*. Half-maximal inhibition of growth was attained at 100 µg/ml with carvacrol while total inhibition of growth was attained with 250 µg/ml citronellol.

Additional Phase I dose-response experiments were also performed to more thoroughly evaluate the efficacy of the β -ionone derivative, β -cyclocitrylidene acetic acid (β -CAA). One hundred µg/ml β -CAA was found to be the minimal inhibitory dose against *B. coagulans* over the 24-hour duration of these experiments. However, it is still unclear whether β -CAA acts primarily as a bacteriostatic or bactericidal agent even though a drop in the OD₆₀₀ was noted after 24 hours of exposure to 150 µg/ml.

2. Phase III Screens:

It was previously reported that 300 µg/ml was the minimum inhibitory concentration of β -CAA when tested in 5-liter fermentors using fresh, production SEL incubated at 42°C (a temperature which is somewhat lower than the average operational temperatures of the production lines at Park 500). To examine this further, an experiment was conducted to see if the

minimal inhibitory dose of β -CAA could be lowered by applying this compound under conditions which more closely resemble those of the upstream portion of the Park 500 process. In pursuit of this, various doses of β -CAA were added to SELs pre-heated to 55°C and then incubated at 42°C in laboratory fermentors to encourage spoilage. The results demonstrated that spoilage of the SELs pre-heated to 55°C could be inhibited by 150 μ g/ml β -CAA over the first 24 hours of incubation while 300 μ g/ml were required previously.

C. Conclusions: None to be reported at this time.

D. Plans:

1. Continue Phase I screening of the appropriate preservative candidates.
2. Determine the minimal inhibitory concentrations for carvacrol and citronellol in the Phase I shake-flask assay.
3. Resolve the mode of action of β -CAA in additional time course studies.
4. Continue evaluation of prospective preservatives in the SEL-based Phase III fermentor assay.
5. Determine the process conditions under which application of a preservative results in optimum efficacy.

E. References:

1. Tenhet, S. Notebook No. 8281, pp. 111-114.
2. Teng, D. Notebook No. 8788, pp. 38, 39.

II. TOBACCO MICROBIOLOGY

A. Objective: To develop methods and to evaluate the microflora resident in tobacco materials.

B. Status:

1. Project ART:

Nine liquid samples obtained from the ART process water scrubber were submitted by Dick Howe for microbial analysis. The samples were plated, incubated and analyzed using the standard operating procedures employed in this laboratory. No bacteria, yeast or mold were observed after 24, 48, 72 and 96 hours of incubation. A memo documenting these findings is currently in preparation.

C. Conclusions: ART process water from the scrubber does not contain bacteria, yeast or mold after 4 days of incubation at optimal growth temperatures.

D. Plans:

1. To submit a proposal to study the microbiological characteristics of selected dry flavors.
2. To continue to develop procedures for the use of the Bactometer® for the quantitation of bacteria in tobacco materials.
3. To support an evaluation of the microbiology of four ART model systems stored under jungle (32°C/85% RH) and desert (43°C/15% RH) conditions.

E. References:

1. Jones, J. Notebook No. 8590, p. 109.

PROJECT NUMBER: 1904
PROJECT TITLE: Tobacco Biochemistry
PROJECT LEADER: D. J. Ayers
WRITTEN BY: V. S. Malik
PERIOD COVERED: April, 1989

I. LOW NICOTINE STUDY

- A. **Objective:** To investigate the biochemistry of the nicotine biosynthetic pathway at the putrescine N-methyltransferase (PMT) step and specifically to isolate PMT from tobacco root extracts.
- B. **Status:** Preliminary studies were conducted to determine the change in the PMT activity of tobacco root harvested at various times after topping. Fifty-two Burley 21 tobacco plants from Group 17 were topped on 3/21/89. Thirteen of the 52 tobacco plants were harvested directly after topping for the T_0 sample. The remaining plants were divided into three groups of thirteen plants and harvested 24 hours, 48 hours, and 168 hours after topping. The specific activity of the roots after topping as compared to T_0 revealed no real changes for the T_{24} sample and increases of 2 and 3 fold for T_{48} and T_{168} , respectively. Leaves (top, mid-stalk and bottom) harvested at various times after topping have been frozen at -80°C to determine if they contain any PMT. All the roots from group 17 of tobacco plants have been harvested and processed to obtain ammonium sulfate (40-65%) extracts (1).

Two 100 ml aliquots from a phenyl-Sepharose column (2) were processed through the S-adenosylhomocysteine (SAH) affinity matrix column (blocked with mercaptoethanol) for further purification of PMT. PMT was eluted from this column with 1 mM S-adenosylmethionine (SAM). The PMT active fractions from the SAH columns were stored at -80°C (3).

Various other PMT active samples and PMT active fractions which did not bind to various SAH columns were concentrated using a DEAE-Sepharose column. Active PMT fractions were bound to the column in dialysis buffer and eluted with 0.2 M NaCl in dialysis buffer (2). These samples were stored at -80°C for use with HPLC.

The sample concentrated by DEAE Sepharose was used to determine the molecular weight of PMT using a TosoHaas PWXL-4000 gel permeation column. An apparent molecular weight between 60 and 70 kD was derived for PMT as calculated by W. P. Hempfling (4). The previous reported MW based on analysis on a silica matrix was lower presumably due to the influence of the salt concentration (3,4).

Using the DEAE concentrated sample (3), a FPLC chromatofocusing column (HR5/5) was examined for fractionation of PMT. PMT activities were eluted at pH 5.1 and pH 5.2 using shallow and steep pH gradients, respectively (4). This is supportive of the earlier determination that isoelectric point of PMT is 5.1 ± 0.1 . The Supelco (C-4 and LC Hint) hydrophobic interaction columns were also examined but deemed to be of little value since PMT activities were

spread over many fractions. However, a Poly LC polypropyl aspartamide hydrophobic interaction column appears promising. The PMT activity can be bound to the polypropyl aspartamide gel in presence of 1 M $(\text{NH}_4)_2\text{SO}_4$ and eluted with about 0.5 M $(\text{NH}_4)_2\text{SO}_4$ (3,4).

As compared to the 2 cm, the 1 cm diameter gel with ELFE looks promising for collecting active PMT fractions. Various parameters with ELFE have been examined. Maximum protein loading capacity on a 1 cm diameter ELFE gel is being determined. Simultaneous runs in which two identical ELFE's, except for the presence or absence of a stacking gel, revealed similar banding patterns of the active PMT fractions. However, the active PMT off the non-stacking gel ELFE eluted in fraction six while the active PMT with the stacking gel eluted in fraction sixteen. Furthermore, there was less tailing observed in the non-stacking gel (5).

The routine Bio Rad Assay was modified to determine protein concentrations in dilute samples. The modification made was to use a large sample volume so that more proteins will be present in the sample to be detected by the dye. The concentrated dye solution is diluted using the sample as the diluent instead of distilled water as in the routine method. Therefore, it is possible to increase the sample volume from 100 μl to 900 μl . Using the modified method, the standard curve constructed using gamma globulin agreed well with that obtained from the routine method. An SAH sample was used to evaluate the validity of the modified method. Accurate determination of the protein levels were seen for SAH samples diluted up to approximately 100 fold (4-5 $\mu\text{g/ml}$); however, when the SAH sample was diluted 200 fold to approximately 2-3 $\mu\text{g/ml}$, the determination was not accurate (6).

- C. Plans: Continue to purify PMT samples from the ammonium sulfate extract through phenyl-Sepharose, SAH, and AEA columns. Implement further protein purification using an HPLC scheme. Explore various conditions using the Rotofor and native gel and ELFE systems. Complete proposal on differential hybridization. Continue to evaluate the modified Lowry method.

D. References:

1. Lyle, J. Notebook No. 8397.
2. Malik, V. Notebook No. 8724.
3. Mooz, E. Notebook No. 8599.
4. Nakatani, H. Notebook No. 8384.
5. Davies, S. Notebook No. 8761.
6. Yu, T. Notebook No. 8806.

PROJECT NUMBER: 2106
PROJECT TITLE: Cigarette Performance and Design
PROJECT LEADER: R. W. Dwyer
PERIOD COVERED: April, 1989

CIGARETTE DESIGN MODELLING (R. Dwyer and J. Kao)

- A. **Objective:** Develop computer programs to assist in designing cigarettes.
- B. **Results:** A system of Fortran-based programs has been developed which allows users to design prototype cigarettes based on the performance of a control cigarette. We are now extending the sytem of programs to handle different filter materials. Our recent efforts have also been in (a) restructuring the programs preparing for further expansions to broaden the capabilities of the system, (b) increasing modularization and documentation of the programs for easy maintenance, (c) improving the speed of computer response time, and (d) improving the friendliness of the user-interface.
- C. **Plans:** Development of new models and enhancement of old models will continue in order to extend the scope of existing models. Integration of these new models into our Fortran-based system of programs will enable us to address the immediate needs in cigarette design. The Fortran-based system shall lay the cornerstone for successful AI applications of cigarette design.

PROJECT NUMBER: 2500
PROJECT TITLE: Fundamental Chemistry
PROJECT LEADER: J. I. Seeman
PERIOD COVERED: April 1989

I. REMOVAL OF NICOTINE FROM AQUEOUS TOBACCO PROCESSING FLUIDS (Howe, Secor, Seeman)

- A. Objective: To develop techniques to remove nicotine and other tobacco alkaloids from aqueous tobacco processing fluids to the exclusion of all other components.
- B. Results and Plans: We have confirmed that BIS is transferred from the organic solvent phase to the feed stock, thereby essentially ruling out its use in any membrane process. Exceptional results have been obtained using our latest synthetic LIX (8764-42B) in the membrane process. High nicotine extraction efficiencies have been noted along with excellent reproducibilities and no phase transfer/breakdown. A UV monitor for the on-line quantification of nicotine has been developed by K. Koller and D. Leyden and installed in our system; it is now being used to excellent advantage. An additional batch (80 g) of LIX(8764-42B) has been prepared and 35 g transferred to Sepracor.

II. INORGANICS AS NOVEL TOBACCO MATERIALS ADDITIVES (Fournier, Paine, Seeman)

- A. Objective: To develop inorganic materials for novel applications for reduced sidestream and enhanced subjectives in cigarettes and for required properties in novel smoking materials.
- B. Results and Plans: We have reproduced Don Schleich's procedure for making a magnesium hydroxide "suspension": a 'slurry' of a white precipitate has been obtained; this material will be submitted for x-ray analysis to confirm the presence of the hydroxide and the absence of oxide. A possible hydrotalcite:vanillin complex has been obtained which incorporates up to 42% of the organic material. This may serve as a novel sidestream reductant and flavor release agent. Preliminary studies have been completed on the solubility of "magnesium carbonate" in carbonated water. Only about 1 g of solid residue was found per 100 g of solution, less than the reported literature values.

III. FLAVOR/ODOR CHEMISTRY (Houminer)

- A. Objective: To develop new technologies for smoke deliveries of desired flavorants, especially menthol.
- B. Results and Plans: The commercial scale manufacturing process for glucose menthol carbonate (GMC) has been reviewed. The key step, the reaction of glucose with menthyl chloroformate was carefully

examined by NMR to determine parameters for the industrial scale-up of GMC. Several potential sidestream release agents have been outlined.

IV. CHEMICAL PHYSICS STUDIES OF TOBACCO CONSTITUENTS (Secor, Seeman)

- A. Objective: To obtain structural information about important tobacco constituents/flavorants; to develop information on cluster formation and chemical reactions in clusters.
- B. Results and Plans: 2-(2-Hydroxyethyl)pyrazine was prepared for examination on its unimolecular decomposition in the gas phase and sent to E. R. Bernstein. This is the parent pyrazine-aldehyde release agent. Current studies at CSU are focusing on the addition of organic acids to allylbenzene and related tobacco flavorants.

V. MISCELLANEOUS (Secor)

- A. Results and Plans: A CR-sheet has been completed for the O-succinylated 4'-hydroxyethylnicotine, prepared for B. Davies. Stock solutions of 10 different nicotinoids were prepared also.

PROJECT NUMBER: 2501
PROJECT TITLE: Smoke Chemistry
PROJECT LEADER: R. Comes
PERIOD COVERED: April, 1989

I. SIDESTREAM SMOKE

- A. Objective:** Conduct studies on sidestream smoke including: development of methods for collection and analysis of sidestream gas phase and semivolatiles; visibility determinations; analysis of selected materials relating to sidestream odor and irritation; development of proprietary products.
- B. Status:** 1.) A number of gas phase runs have been made utilizing the Tekmar desorber under varying conditions to attempt to establish an optimal standard operating procedure. Changes have been studied varying both sample collection procedures and gc temperature program conditions. Problems with plugging of the desorber continue to be a significant cause of system down time. Development of this methodology will continue. 2.) Efforts to correlate subjective attributes of gc gas phase peaks have continued. A chromatogram was produced using the PE LIMS system. Peak retention times and mass spectral identifications were included. Comparison of the chromatogram with odor profiles indicated that the compounds identified were not those responsible for the intense odors detected. To address this finding, a more sensitive sulfur detector has been ordered to study possible differences in the profiles of the gas phase sulfur compounds. 3.) Routine BET surface area measurements and single port visibility determinations continue to be run as requested. A discrepancy between the extinction measured by the computer and the extinction shown on the chart paper was noted with the single port apparatus. Various system checks tend to indicate the problem is associated with electrical line noise. This situation is being addressed. 4.) The second 8-port smoking machine has arrived. All components for set up of this second visibility apparatus are either on-hand or under construction. Modifications to the first 8-port system continue as needed in order to make this system as operator "friendly" as possible. Certification of this system utilizing United States Testing Co. is currently underway. A series of calibrations and test runs will be made in support of this certification procedure. 5.) A significant project effort was directed at modification and operation of the CORESTA sidestream smoking machine. This apparatus was originally designed to smoke conventional diameter, 85mm cigarettes. In order to smoke cigarettes of 100mm length and/or 17mm diameter, new chambers were designed and built. These larger chambers required significant modifications to the sidestream air flow which then required the installation of a new flow monitoring system and several related alterations to the overall apparatus. Once acceptable conditions (based on correct MSTPM and static burn times for controls) were established, a series of six cigarettes (Monitor-25, Vantage Excel, Marlboro Light 100's, B&H 100's, Trim X and Capri) were smoked. Sidestream TPM, nicotine and water data

were obtained under both static and dynamic smoking conditions. Comparable mainstream data was generated during the dynamic smokings. A report is currently being written. These cigarettes are also being studied for visibility using the single port visibility apparatus. 6.) The program to measure the condensable build-up in a burning cigarette continues using neutron radiography. Several runs have been made using varied models. The large amount of data generated in these runs is presently being analyzed.

II. SIDESTREAM SMOKING CHAMBER

- A. Objective: Design and construct an environmentally controlled chamber to measure selected components of sidestream smoke.
- B. Status: 1.) A visit was made to the facility where the chamber is being constructed. Pending some relatively minor modifications, the chamber was accepted and should be delivered to P.M. on May 1. Installation and check-out should require two weeks. 2.) Additional chamber instrumentation has been ordered to facilitate analysis of some other components.

III. MISCELLANEOUS

1. A series of cigarettes to determine nicotine delivery to sidestream is being smoked on the CORESTA apparatus. Seven models with varied levels of nicotine added to ART filler have been made and will be evaluated for sidestream TPM, nicotine, water and static burn time. The control is a zero add-back to the ART filler.
2. Thirty-four samples were analyzed for nicotine in support of the Sepracor program.
3. Multiple samples have been analyzed by gc/ms and pyrolysis/gc/ms as requested.
4. Neutron activation analyses continue to investigate the potential transfer of aluminum to smoke in control vs sol-gel treated cigarette papers. Condensate from four experimental cigarettes (IR4F, D9UR, D9US and D9US-1) was collected directly into acid washed vials. The condensate was activated and then transferred to non-irradiated vials to eliminate vial activity contamination. Data evaluation is underway.
5. Various CORESTA sidestream chambers were examined with schlieren, visual and infrared cameras in conjunction with J.J. Piade from FTR. The flow of the smoke in the different chambers with different draft velocities was investigated with the schlieren (did not work due to interference from the chamber walls) and the visual cameras. The infrared camera work was to examine the effect of the draft velocity in each chamber on the coal temperature distribution. This data has yet to be analyzed.

6. Section and total distribution data have been obtained on the labelled cigarettes prepared in support of the Biochemical Research Division nicotine precursor/product study. These results have been summarized in a memo.

PROJECT NUMBER: 2520
PROJECT TITLE: Flavor Research
PROJECT LEADER: E. W. Southwick
PERIOD COVERED: April, 1989

I. GLUCOSE-DERIVED FLAVORANTS

- A. Objective:** To develop glucose-derived flavorants.
- B. Results:** 1.) Nearly 100g of glucovanillin has been provided to Flavor Development to support their studies of sidestream odorants. An additional 300g is in the final stage of preparation. Additional quantities will be available from FD&O. 2.) A small amount of gluco-ethylvanillin was prepared for analytical and subjective studies. 3.) Plans have been formulated and synthesis initiated to obtain glucose-derived sidestream odorants for application to paper.

II. PROJECT EXTRA

- A. Objective:** To develop proprietary flavor additives for enhanced flavor perception in low delivery cigarettes.
- B. Results:**
- 1.) Oxalate ester release agents
 - a. Evolved gas analysis of 4-oxo- β -ionyl methyl oxalate showed carbon dioxide as the only gaseous component of pyrolysis. TGA showed the onset of decomposition at about 205°C. This compound cleanly pyrolyzes to give megastigmatrienones.
 - b. Methyl oxalate esters of pyrazinyl alcohols were pyrolyzed at 300°C in a flash vacuum thermolysis apparatus. The following unsaturated pyrazines were isolated in high purity:
 1. Isopropenylpyrazine
 2. 2-Isopropenyl-3-methylpyrazine
 3. 2-Isopropenyl-3,6-dimethylpyrazine
 - 2.) Hexahydro-2(3H)-benzofuranones (sweet flavor)
 - a. CR-2643 (6-methylhexahydro-2(3H)-benzofuranone) is a component of a flavor formulation currently in POL testing. The natural product, cis-3,6-dimethyl-3a,4,5,7a-tetrahydro-2(3H)-benzofuranone has been synthesized and found to have very similar odor properties (almond, minty, sweet, coconut) to CR-2643.
 - b. Synthetic methods to prepare 6-substituted hexahydro-2(3H)-benzofuranones have been developed. The synthetic sequence was demonstrated by the preparation of 6-t-butylhexahydro-2(3H)-benzofuranone in 50% overall yield.

c. The unsubstituted analog of the natural product described above was prepared in 50% overall yield. Structure-odor relations for all these compounds continues to develop a receptor-site map for sweet flavor.

III. TOBACCO-IDENTICAL FLAVOR

- A. Objective: To identify tobacco components which are sensorially significant.
- B. Results: The roasted volatiles of burley tobacco were generated by several modifications of the original procedure. Based on earlier experience, the portion of the basic fraction eluting prior to nicotine in the gas chromatogram was most closely investigated. Components were trapped and evaluated on 100% DIET cigarettes with the following subjective results:
1. total section prior to nicotine: increased body/fullness, green, bitter, some roasted notes.
 2. sub-sections of above
 - a. sharp/pungent, nutty, musty
 - b. heavy, dark, dirty, green
 - c. toasted, woody, green
 3. sub-section of (2b)
 - a. musty, dirty, bitter, high lung impact
 - b. coffee, roasted, green/bitter, increased body

GC/MS analysis showed the presence of 24 pyridines, 29 pyrazines and 11 thiazoles, many of which have not been previously reported in tobacco.

PROJECT NUMBER: 2525
PROJECT TITLE: Tobacco Chemistry
PROJECT LEADER: R. R. Izac
PERIOD COVERED: April, 1989

I. NATURAL PRODUCTS CHEMISTRY

- A. Objective: To isolate, identify and/or analyze natural compounds with major emphasis on tobacco and tobacco products.
- B. Results: An HPLC procedure for the analysis of nicotine and the minor alkaloids is being optimized for the analysis of Nicotiana species. Standard curves were optimized for nicotine and nornicotine (R = 0.999 and 0.999 respectively) in the region of 1.1 to 4.1 μ g.
- C. Plans: Continue to optimize modified procedure.
- D. References:
 - 1. Izac, R. Notebook No. 8632.
 - 2. Core, M. Notebook No. 8608.

II. GREENHOUSE STUDIES

- A. Objective: To maintain the R&D greenhouses, to conduct plant research studies and to provide greenhouse-grown tobacco materials for support of other R&D programs.
- B. Results: The hydroponic Burley 21 plants in Group 17 were harvested. A total of 8629g of fresh root tissue was obtained for Project 1904. Transplanting of Group 18 plants was completed.
- C. Plans: Maintain production of fresh root tissue by hydroponic culture.
- D. References:
 - 1. Bass, R. Notebook No. 8787.
 - 2. West, G. Notebook No. 8559.
 - 3. Newell, G. Notebook No. 8762.

III. SUPPORT ACTIVITIES

- A. Objective: To provide requested assistance for special projects.
- B. Results: 1.) A sample of β -cyclocitrylidine acetic acid was hydrogenated. This compound is being evaluated as a substitute for paraben. 2.) Hand fabricated cigarettes (about 224) were prepared for the LOTUS program. 3.) A total of 12 14 C-nicotine cigarettes were hand fabricated for the TSNA program. 4.) A tartaric acid derivative was purified for the Sepracor project.

C. References:

1. Izac, R. Notebook No. 8632.
2. Bass, R. Notebook No. 8787.
3. Newell, G. Notebook No. 8762.

PROJECT NUMBER: 6502
PROJECT TITLE: Environmental Tobacco Smoke
PROJECT LEADER: C. E. Thomas
PERIOD COVERED: April, 1989

I. MAINSTREAM AND SIDESTREAM SMOKE STUDIES

- A. Objective: Determine if the methanol measured in an unlit puff of a production marlboro is due to solvents in pack materials.
- B. Results: The tunable diode laser spectroscopy system was used to evaluate the levels of methanol in unlit puffs from Marlboro 85mm and 80mm cigarettes (manufactured at MC, March, 1988). There were no differences in unlit puff methanol of cigarettes, which had been analysed directly from the pack, with cigarettes that had been collected loose at the makers.
- C. Conclusions: In the samples that were investigated, methanol in the unlit puff and in MS smoke is not originating from solvents used in packaging materials
- D. Plans: Samples will be heated in a 55°C oven for 24 hours to determine if free methanol may increase due to reactions taking place within the tobacco or flavoring systems. The Marlboros studied initially will be evaluated for changes in the free menthanol as a function of cigarette age.
- E. References:
1. Lipscomb, J., Notebook 8703, pp. 78, 83.
 2. Parrish, M. Notebook #8729, pp. 33-44.

II. PROJECT ART

- A. Objective: Evaluate the effect of addition of selected acids on the ammonia delivery of ART cigarettes.
- B. Results: Machine-made ART cigarettes (D9TC-1) were used in several studies in which either citric or lactic acids were injected on the filler or filters. These additions resulted in reductions in MS ammonia deliveries with the highest reductions observed when the acids were placed on the filters.
- C. Conclusions: The addition of citric and lactic acids reduce the ammonia deliveries of ART cigarettes. In one specific experiment, a 55% reduction was observed in the MS gaseous ammonia when 2 mg of citric acid was added to the cigarette filter.
- D. Plans: The experiments will be repeated using 5 mg each of malic, citric, and lactic acid on the filters of the ART cigarettes. Additional MS total ammonia determinations and TPM fractionation data will be used to study changes in the basicity of the MS smoke.

E. References:

1. Parrish, M., "Summary of Results and Recommendations for the Reduction of MS Ammonia in Art Cigarettes", Memo to J. Charles, April 12, 1989.
2. Parrish, M., White, G., "Reduction of Total Ammonia MS Delivery in ART Cigarettes Treated With Lactic Acid", Memo to C. Kroustalis, April 12, 1989.
3. Parrish, M., PM Notebook 8729, pp. 28-29.

III. ETS STUDIES

- A. Objective:** Provide analytical support to project PACT by measuring the various constituents of environmental tobacco smoke.
- B. Results:** In Phase I of the studies being conducted by project PACT, ventilation, Electrostatic Precipitation (ESP), and selected carbon filters have been evaluated for the removal of particulates and nicotine. Currently sixteen different experiments have been completed. For each experiment six PASS cases are used to measure the total particulates (RSP), nicotine, CO, temperature and barometric pressure. Additional instrumentation is employed to measure the distributions in the test room (175 m³) of particulates, CO, CO₂, temperature, and relative humidity. In a control experiment, the RSP was 1.8 mg/m³, CO was 13 ppm, and the nicotine was 0.56 mg/m³ when ventilation (3.5 air exchanges per hour) was the sole source for smoke removal. When an ESP unit was added with an additional recirculated volume of air of 17.5 m³/minute, these values were reduced to 0.64-mg/m³ RSP, 0.47 mg/m³ nicotine, and 11 ppm CO.
- C. Conclusions:** The ESP unit has been shown to remove particulates from room air as well as providing some reduction in the room nicotine concentrations. The limiting factor in the reduction of smoke particulates is the effective ventilation rate in air exchanges per hour of the ESP unit.
- D. Plans:** This project will continue to provide analytical support for project PACT. Additional tests will be conducted to study the removal efficiency of different ESP units for particulates. In Phase II of this study methods will be developed to study the reduction of gas phase components of cigarette smoke.
- E. References:**
1. Lipscomb, J., Notebook #8703, pp. 88-90.
 2. White, G., Notebook #8688, p. 43.
 3. Koller, K. Notebook #8700, p. 70

PROJECT NUMBER: 6505
PROJECT TITLE: Special Investigations/Methods Development
PROJECT LEADER: D. F. Ingraham
PERIOD COVERED: April, 1989

I. PROJECT ART

- A. Objective: Provide analytical support to project ART.
- B. Results: The precision of the new nicotine method (E-86A) was determined using ground monitor samples. The two monitors used were uncased ART extracted filler and a DL-5 blend. Two extracts from each monitor were analyzed every day for 10 days. The relative standard deviations were 5% and 3% for the extracted and unextracted monitors, respectively.
- C. Plans: Continue providing analytical support to the BHPP and ART production facility during start-up on an as-needed basis. Begin an inter-laboratory study on April 24th to determine if there are any differences between nicotine results using Method E-86A from the Bermuda Hundred Processing Facility and Analytical Research.

II. ANALYSIS OF RESIDUAL SOLVENTS IN PACKAGING MATERIAL

- A. Objective: To provide headspace analyses for residual solvents from packaging materials.
- B. Results: A study was begun to qualify four of our printers in the use of the headspace method for residual solvents. Three solvent mixtures and a standard mixture were prepared and given to J. Stargardt for distribution to the vendors and QA. No results are available at this time.
- C. Plans: We will continue to analyze non-routine samples and provide support to QA as needed, including further training of the analyst in QA. Analyze the results from the vendors.

III. MATERIALS EVALUATION

- A. Objective: To identify components of commercial products prior to their use at PM facilities.
- B. Results:

Samples analyzed this month included conveyor belts, labels, gloves, and cleaners. Samples of adhesives, lacquers, and headspace volatiles from packaging were analyzed for ammonia for Packaging Development.

A presentation on the Materials Evaluation program was made at the TOAF facility and at 20th Street for plant management. At the Quarterly Materials Evaluation Committee meeting a list of 175 items from the Materials Evaluation REDBOOK for which recommendations were changed was distributed.

- C. Plans: Materials Evaluation is an on-going program.

IV. ELEMENTAL ANALYSIS

- A. Objective: To provide qualitative and quantitative elemental data on tobacco, cigarette paper and material evaluation samples.

B. Results:

Numerous paper samples were analyzed by x-ray fluorescence for S. Baldwin and B. Goodman to determine elemental concentrations, primarily Mg, K, and Ca. These analyses included the determination of which side of the paper additives (in particular Mg) had been applied. Results from these papers will be added to the tables being used for evaluation of the new paper method.

Thin film standards are being evaluated for use as standards for the paper method. These are NBS certified polycarbonate films that have single elements or multiple elements applied to them.

Numerous routine tobacco samples were analyzed this month. These included high K stems, sheet material and treated filler.

Visits were made to three ICP vendors to evaluate instrumentation for use at PM. These samples were digests of previously analyzed flavor samples in ethanol. Data were collected for comparison to determine the best ICP for our needs.

- C. Plans: Evaluation of the paper method will continue. Data from the three ICP's will be evaluated and a recommendation made.

V. RESPONSE TO ANALYTICAL REQUESTS

- A. Objective: To provide analytical support to R&D and Operations personnel and projects.

B. Results:

Analyses and investigations by project personnel during the month of April included:

Menthol, triacetin, and water determinations in plugs were provided for T. van Auken. This was part of a study to determine if any relationships existed between moisture and triacetin levels and the amount of menthol adsorbed. The results were reported to Dr. van Auken for analysis.

Ethylvanillin glucoside was confirmed as a paper additive in Chelsea cigarettes. This compound releases ethylvanillin to sidestream upon smoking. The glucoside was present at a level of approximately 0.25% on the paper, based on a synthetic standard

supplied by G. Chan. The scratch-and-sniff sample on the top of the pack was found to contain ethylvanillin.

Nicotine levels and volatile organic profiles were provided to E. Thomas and J. Lephardt as part of an environmental tobacco smoke study currently in progress.

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PROJECT NUMBER: 6902
PROJECT TITLE: Biochemical Special Investigations
PROJECT LEADER: B. D. Davies
WRITTEN BY: E. A. Crockett
PERIOD COVERED: April, 1989

I. NICOTINE SPECIFIC MONOCLONAL ANTIBODY

A. **Objective:** To obtain a monoclonal antibody (MCA) against nicotine (NIC-MCA).

B. **Results:** Sera were analyzed for the presence of specific classes of immunoglobulins using an anti-IgG (AIgG) and an anti-IgM (AIgM) screening antibody. Using these antibodies the titers for each class were determined. It appears that the dominant nicotine specific antibodies in sera are of the IgG class.

Several Enzyme-Linked Immunosorbent Assays (ELISAs) were run using AIgG to test the reactivity of the polyclonal sera (PCA) with 10 nicotine analogs supplied by J. Seeman and H. Secor. The compounds which were used in the inhibition assay were d,l-nornicotine, anabasine, d,l-anatabine, nicotinic acid, continine, trans-nicotine-n-oxide, 2-3'-dipyridyl, nicotyrine, myosmine, and N'-methylanabasine. With the exception of N-methylanabasine, none of the compounds exhibited greater than 20% inhibition when tested at a 1×10^{-4} M concentration indicating no interference. When examined in an extended test, N-methylanabasine was found to inhibit the PCA in a dose dependent manner with a 50% inhibition constant (IC₅₀) of 7.9×10^{-5} and 1.0×10^{-5} for the two PCAs tested. In the same experiment, nicotine displayed an IC₅₀ of 1.6×10^{-4} and 4.0×10^{-5} , respectively.

Initial screenings of spent culture media, using AIgG, did not show a positive antibody response. Subsequent screens have demonstrated that antibodies are present and are of the IgM class. This has been confirmed by Litron Laboratories. However, even when using AIgM the culture media showed no dose response to nicotine.

Other assays were performed to examine the possibility of reducing the time required to perform the assay. The following results were observed: the plates can be prepared the morning of the experiment as opposed to a 16 hour pre-incubation; the blocking period can be reduced from 1 hour to 5 minutes; but the incubation periods for the primary and secondary antibody must remain at least 2 hours.

C. **Plans:** Instruct Litron to continue with the fusion and test spent culture media from selected clones.

D. **References:**

Davies, B. D. Notebook No. 8638, p. 185
Crockett, E. A. Notebook No. 8783, p. 65

2022172541

II. ADDITIONAL APPROACHES TOWARD PUTRESCINE METHYLTRANSFERASE (PMT) ISOLATION

- A. Objective: Provide additional experimental approaches to assist in the effort to isolate PMT.
- B. Results: A sample of M5-q-SAH, material obtained from E. Mooz was concentrated and photoaffinity labeled. The labeled sample was fractionated using 1D-SDS polyacrylamide gel electrophoresis, electroblotted, and specifically labeled PMT peaks identified using the RLTC scanner. Preliminary results indicate a 4:1 incorporation of radioactivity in the total sample compared to the non-specific sample. The RLTC scan showed one highly radioactively labeled peak. The molecular weight is currently being determined for this peak.
- C. Plans: To complete the calculations necessary to determine the molecular weight of the peak. In addition, perform a time course study on the photolysis procedure in order to minimize light damage to the proteins.
- D. References:

Davies, B. D. Notebook No. 8638, p. 185
Dunn, R. Notebook No. 8721, p. 55-80

PROJECT NUMBER: 6906
PROJECT TITLE: Biological Effects of Smoke
PROJECT LEADER: J. M. Penn
WRITTEN BY: G. J. Patskan
PERIOD COVERED: April, 1989

I. GLUTATHIONE DEPLETION ASSAY (GDA)

- A. Objective: To determine the source of inconsistencies in GSH levels and GSH/GSSG ratios in TA98 cells.
- B. Results: In one experiment the levels of GSH and the GSH/GSSG ratios were evaluated in: the current stock culture of TA98, a stock culture of TA98 prepared in 1986 and the current stock culture of TA100. Each culture had little to no GSH present. Another experiment compared the current lot of growth medium with a new lot. Again only low levels of GSH were detected. In two additional experiments the source of the deionized water, the sonicator and the trichloroacetic acid were evaluated. None of these factors appeared to be the source of the low GSH levels. However, the GSH levels and the GSH/GSSG ratio had returned to about 50% of normal levels.
- C. Conclusions: The reason for the aberrant GSH and GSSG values has not been deduced.
- D. Plans: The work utilizing TA98 cells will be put on hold in favor of examining the effects of indomethacin and phospholipase inhibitors on the GSH levels in CSC-treated V79 cells.
- E. References:

McCoy, W. R. Notebook No. 8739, pp. 78.

II. 3T3 CELL CULTURE MAINTENANCE

- A. Objective: Establish and maintain a 3T3 cell culture for use in biochemical assays.
- B. Results: The new 3T3 cell culture is being maintained using the previously established seeding protocol as well as a modified protocol with lower seeding densities. Cells grown according to the modified protocol have been tested in EGF (1) and PDBu binding (2) experiments. One EGF binding experiment demonstrated lower EGF binding in untreated cells and greater sensitivity to 2R1 CSC than the previous 3T3 cell culture. Several PDBu experiments were conducted with results similar to those obtained with the previous 3T3 cell culture. Additionally, a soft agar growth experiment was conducted which indicated that the current 3T3 cell culture lacks the ability to grow in semisolid medium (3).
- C. Conclusions: The current culture of 3T3 cells can be used in the EGF and PDBu binding assays. Also, the current culture of 3T3

cells appears to display characteristics which have been reported in the external literature.

- D. Plans: Continue to maintain the 3T3 cells using both the modified and previous protocols. If significant differences in the growth characteristics develop between the cells passaged by each method further comparisons will be made.

E. References:

1. Stagg, D. L. Notebook No. 8553, p. 151.
2. Burruss, T. J. Notebook No. 8804, p. 48.
3. Penn, J. M. Notebook No. 8217, p. 95.

III. ACQUISITION AND MAINTENANCE OF ADDITIONAL CELL LINES

- A. Objective: To acquire and maintain a variety of cell lines for use in biochemical assays.
- B. Results: The 3PC mouse keratinocyte cell line has been cryopreserved and a sample has been prepared for mycoplasma testing (1). The MT1/2 mouse keratinocyte cell line was lost due to fungal contamination (1). Each of the JB-6 cell clones has been cryopreserved (2). Evaluation of three lots of serum for their ability to maintain the growth of the JB-6 cells has begun (2).
- C. Conclusions: These cell lines are being maintained with characteristics similar to the literature.
- D. Plans: Obtain a new culture of MT1/2 cells from the University of Texas. Continue the characterization of the other cell lines.

E. References:

1. Patskan, G. J. Notebook No. 8751, p. 188.
2. Burruss, T. J. Notebook No. 8804, p. 48.

IV. PROTEIN KINASE C (PKC) ASSAY IN INTACT CELLS

- A. Objective: To examine the response of 3T3 cells to 12-O-tetradecanoylphorbol-13-acetate (TPA) in the PKC intact cell assay, using the most recent 3T3 cell culture in both log and quiescent phase.
- B. Results: Several modifications were made in the assay: TPA was present in the labeling medium, autoradiographs of PAGE gels were scanned on the densitometer perpendicular to the direction of the lanes of treatment, i.e., holding the molecular weight constant, and the peaks from these scans were integrated. TPA treatment seemed to result in greater increases in the phosphorylation of a 71 kDa molecular weight protein than other proteins. Quiescent cells appeared to have lower basal levels of protein phosphorylation than log phase cells.

- C. Conclusions: The modified method of data analysis appears useful in detecting differential phosphorylation of certain proteins when subjected to TPA treatment.
- D. Plans: Repeat PKC assay testing TPA and 3 CSCs (2R1, X6D4CDL and X6D5RN) at two concentrations using the the new treatment protocol and new method of data analysis. Determine the growth rate of cells in culture after growing in conditions designed to induce quiescence.
- E. References:
Nixon, G. M. Notebook No. 8711, p. 70.

V. CHARACTERIZATION OF JB6 MOUSE EPIDERMAL CELL LINE

- A. Objective: To determine if the JB6 cell line will form colonies in a semisolid medium.
- B. Results: One assay was conducted according to the literature protocol (Difco agar, 10% Fetal Bovine Serum) using the three different clones. No colony growth in soft agar was observed for any of the clones.
- C. Conclusions: The transformed clone (RT101) should have produced colonies in soft agar without any treatment. The lack of colony production indicates that changes in assay parameters are necessary to produce colonies of RT101 under control conditions.
- D. Plans: Another assay has been started in Noble agar using only RT101 cells in three different sera. The serum content of the medium will be tested at 10% and 20%.
- E. References:
Nixon, G. M. Notebook No. 8711, pp. 57-8, 69.

VI. PDBu BINDING ASSAY

- A. Objective: Determine assay conditions under which CSC will not reduce the nonspecific binding of PDBu to 3T3 cells at 4°C.
- B. Results: A time course of binding experiment was conducted which compared the effects of DMSO and 2R1 CSC from 15 to 180 minutes. PDBu binding appeared to reach equilibrium after only 15 minutes. CSC had elicited reduced nonspecific binding by 15 minutes. In a second experiment the cells were incubated with PDBu until equilibrium was reached then either TPA or 2R1 CSC was added without removing the PDBu solution. TPA reduced specific binding as a function of time with no effect on nonspecific binding. CSC reduced nonspecific binding with little effect on specific binding. In a third experiment the cells were incubated with PDBu

until equilibrium binding was achieved, then the PDBu solution was removed. At this time TPA and CSC were added. PDBu was rapidly lost from the cells with no effect of TPA or CSC on that loss.

- C. Conclusions: None of the changes in the routine assay was satisfactory for evaluating the ability of CSC to bind to the phorbol ester receptor.
- D. Plans: Conduct an experiment in which the cells are first treated with CSC at 4°C followed by the addition of PDBu.
- E. References:

Burruss, T. J., Notebook No. 8804, p. 48.

PROJECT NUMBER: 6908
PROJECT TITLE: Smoke Condensate Studies
PROJECT LEADER: A. H. Warfield
PERIOD COVERED: April, 1989

I. TSNA PRECURSORS

- A. Objective: To determine the precursors of MS TSNA.
- B. Results: Data collection was continued as part of an aging experiment on water-washed burley filler. Time-course filler TSNA and alkaloid data are now available after storage at -30° and 45°C for 21, 42 and 63 days. Thus far there has been little change in endogenous NNN and NAT, but there is a definite trend towards higher NNK in the 45°C sample with time. There has been no change in alkaloid levels (measured by KOH/methanol extraction followed by GC/NPD) found in the filler over the observation period. Only one MS TSNA determination has been made thus far (42 days), and these data show no change in MS NNN or NAT, but there is an apparent increase in MS NNK. More data will have to be collected to determine whether this increase is significant.

Additional evaluations of stored RLs from the Crossed Solubles-Base Web (CSBW) study were carried out. Filler NNN and NAT levels of BuCEL on BuBW increased by approximately 40% after one year at room temperature, while filler NNK increased by 50%. MS NNN and NAT were only slightly higher after the aging period relative to the time immediately after preparation of the RL, but MS NNK was increased by 87%. Similar studies were carried out on the BuBW used to prepare the RL, but the values obtained were much lower than those obtained when the RL was analyzed, and no definite conclusions could be drawn.

- C. Plans: The aging study samples will be analyzed for filler TSNA every three weeks and MS TSNA every six weeks until July, 1989.

D. Reference:

Haut, S. A. Notebook No. 8768, p. 108.

II. ORIENTAL TOBACCO STUDIES

- A. Objective: To determine if the inhibition of TSNA formation/pyro-synthesis observed for oriental tobacco is due to agronomic effects.
- B. Results: Nicotine and minor alkaloid data on eight oriental varieties were obtained by capillary GC/NPD. The analyses were performed by Bob Levins (Project 6912). Total alkaloid data were obtained from ARD on the same samples. There was considerable variation in individual alkaloid levels among the different varieties. Nicotine levels ranged from 0.3% to 2.9%, while normicotine levels ranged from 0.005% to 0.07%.

C. Plans: Filler and MS TSNA data as well as further ARD analyses will be completed on the eight oriental varieties, and the individual varieties will be evaluated with respect to their likely level of inhibition of MS TSNA.

D. Reference:

Keene, C. K. Notebook No. 8754, p. 140.

III. ORIENTAL INHIBITOR STUDIES (CEL)

A. Objective: To determine the causative agent(s) responsible for the reduction in MS TSNA observed when oriental (Or) CEL is added to burley (Bu) CEL and applied to Bu base web (BW), relative to a control RL prepared from BuCEL on BuBW.

B. Results: Studies were continued in which the effect of OrCEL and sugars (present in special RLs) on sidestream (SS) as well as MS TSNA were determined. The test RLs were made from mixed Bu and Or CELs and BuCEL + "sugars" on BuBW. Corresponding controls were made from two levels of BuCEL on BuBW. ("Sugars" consisted essentially of the calculated amounts of individual sugars present in the OrCEL.) Initial results were reported in February. The inhibitory effect of OrCEL on SS TSNA appeared to be much smaller than that on MS TSNA. In addition, an increase in MS TSNA deliveries was noted on aging of the RL containing OrCEL.

Current results show that a two-fold increase in MS TSNA from BuCEL on BuBW occurred during a one year of storage at room temperature after the RL was prepared. Addition of OrCEL caused a lower initial MS TSNA level and greatly attenuated the increase in MS TSNA on aging. Addition of sugars also lowered initial MS NNN and NAT, but (in contrast to OrCEL) resulted in a further decrease in MS NNN and NAT on aging, while there was a slight concomitant increase in MS NNK. The observation (previously reported) that OrCEL caused reduction of MS NNK, whereas sugars had no effect on initial MS NNK, was taken as an indication that OrCEL may contain inhibitors capable of reducing MS NNK. These inhibitors are apparently different from those derived from the sugars contained in OrCEL. There was an indication that the total increase in MS NNK that occurred in the one year period (in the absence of an added inhibitor) occurred in a 1.5 month period, whereas the increase in MS NNN and NAT appeared to be linear with time. Insufficient data are presently available to confirm the latter observations.

SS TSNA data on the above samples are now available after an elapsed time of 1.5 and 7 months since the RLs were prepared. As reported earlier there was an increase in SS TSNA with time, but the slope of the increase was much smaller than that observed for MS TSNA.

Because of the fact that sugars added to BuCEL (which contains high levels of soluble ammonia) have been observed to effect

reductions in MS TSNA, it has been postulated that sugar-ammonia reaction products may be responsible for the reductions. Bright filler contains high levels of reducing sugars but little soluble ammonia. Therefore, ammonia as diammonium phosphate (DAP) was added to bright CEL at a target level of 0.37% ammonia in the RL. (BuCEL + "sugars" on BuBW contained 0.53% ammonia). The control was a bright RL containing 0.06% ammonia. The levels of individual sugars were similar to those in the BuCEL + "sugars" RL except that there was more glucose in the latter. However, addition of ammonia at this level did not affect the levels of either MS or SS TSNA obtained on smoking cigarettes made from these RLs.

- C. Plans: Data collection on the BuCEL/OrCEL/"sugars" on BuBW aging study will continue. An RL has been prepared from mixed Br and Bu CELs on BuBW in order to further investigate the possible contribution of ammonia-sugar reactions or related reactions to the inhibition of MS TSNA observed on addition of OrCEL to the BuCEL on BuBW RL.

D. Reference:

Morgan, W. R. Notebook No. 8579, pp. 158-163.

IV. ORIENTAL INHIBITOR STUDIES (ORGANIC EXTRACTS)

- A. Objective: To determine whether the causative agent(s) responsible for the reduced levels of MS TSNA observed for Or tobacco can be removed with organic solvents and applied to other fillers as a means of decreasing the MS TSNA levels delivered by these fillers.

- B. Results: With the assistance of R. Izac, a blended sample of oriental tobacco was extracted sequentially with methylene chloride (MeCl) and methanol (MeOH). These extracts were applied to two test fillers: burley filler extracted with 5% EtOH/hexane (ExBu), and BuBW. The extracted filler is low in nicotine and endogenous TSNA but still contains minor alkaloids, salts, and other water solubles. BuBW is devoid of all the above but still produces pyrosynthetic NNK. Data obtained thus far have resulted in reductions of 34, 30, and 11% in MS NNN, NAT, and NNK, respectively, for ExBu filler treated with the MeCl extract.

- C. Plans: The remaining samples will be evaluated for MS TSNA deliveries.

D. Reference:

Haut, S. A. Notebook No. 8768, p. 108.

V. MISCELLANEOUS AND SUPPORT STUDIES:

- A. Objective: To conduct studies of the TSNA content of filler and/or MS smoke as necessary to support other PM programs.

B. Results: MS TSNA data were obtained on machine-made cigarettes consisting of RCB and full-flavored blend. The RCB included washed burley stems (control) or washed burley stems combined with Post-ART bright stems. The data have been discussed in a memo.

C. Reference:

Haut, S. A. Notebook No. 8768, p. 108.

PROJECT NUMBER: 6912
PROJECT TITLE: Tobacco/Smoke Relationships
PROJECT LEADER: S. B. Hassam
PERIOD COVERED: April, 1989

I. TSNA PRECURSORS

- A. Objective: To determine the precursors of MS TSNA.
- B. Results: Radiolabeled nicotine cigarettes (code 3-89-3) were smoked on a single port total recovery smoking machine by Dick Newman of the Chemical Research Division (CRD). Two smokings of five cigarettes each were done (4/5/89, 4/12/89). In each set, MS TPM and SS TPM were collected from each cigarette on disposable Cambridge pads. Fresh pads were used for each cigarette. The TPM pads were shaken in citrate/phosphate buffer containing L-ascorbic acid. From each smoking the MS TPM buffer extract was extracted with methylene chloride. The organic phase was washed with 2N sodium hydroxide, dried over sodium sulphate, and excess solvent was removed under reduced pressure. The residue was redissolved in methylene chloride (10 mL).

Liquid scintillation counting (LSC) of the buffer extracts from smoking #1 yielded the following results: MS TPM buffer extract, 6.44 μCi ; SS TPM buffer extract, 10.51 μCi ; SS buffer rinse of smoking chamber, 4.74 μCi . The results for smoking #2 were as follows: MS TPM buffer extract, 6.36 μCi ; SS TPM buffer extract, 9.90 μCi ; SS buffer rinse of chamber, 5.89 μCi . (The estimated initial activity per 5 cigarettes is about 50 μCi , based on values determined by sectioning 2 cigarettes; the radioactivities in the gas phases and in the butts were determined by Dick Newman and the total recovered activity is estimated to be about 90%).

Radioactivities for the methylene chloride solutions obtained from the MS TPM buffer extracts were: smoking #1, 233 nCi; smoking #2, 257 nCi. Preliminary radiochromatography on TLC plates of the MS TPM buffer extracts and methylene chloride solutions indicated that virtually all of the radioactivity is associated with ^{14}C -nicotine.

- C. Plans: Continue radiochromatography of MS TPM extracts. Isolate TSNA by preparative TLC and analyze by radiochromatography and by GC/TEA. Nonradiolabeled cigarettes of the same filler blend and configuration obtained from CRD will be smoked and analyzed for TSNA by Project 6908.
- D. Reference:

Hassam, S. Notebook No. 8712, p. 179.

II. CROSSED SOLUBLES/BASE WEB STUDY (CHEMISTRY)

- A. **Objective:** To investigate the smoke chemistry of model cigarettes made from all possible combinations of solubles from bright, burley and oriental tobaccos on base webs from the three tobaccos.
- B. **Results:** The preparation of fillers using bright base web and burley CEL, each containing different levels of added calcium acetate, was completed. Handmade cigarettes were prepared from each of the 23 filler types. Cigarettes were smoked in quadruplicate, 5 cigarettes per smoking to provide IT CSC. The CSC from each smoking was dissolved in DMSO for S/M assay.

The preparation of fillers using bright base web and bright CEL containing different levels of our standard protein was completed. Fillers were also prepared with bright CEL containing different amounts of an amino acid mixture, and different amounts of ammonium acetate. The preparation of the fillers was done in a collaborative effort with E. Lambert (Project 6908).

Elution of a weakly acidic cation exchange resin previously treated with burley CEL was continued with 0.05M ammonium acetate. This treatment yielded 7.1 g of additional material. A final treatment with 0.05M sodium hydroxide yielded 1.8 g of material. The total recovery of material, including 51.4 g eluted initially with water, and 15.5 g eluted with methanol and methanol/acetic acid, was 46.7% of the burley CEL treated with the resin.

Burley CEL (171.8 g) was treated with an ion retardation resin (Bio-Rad AG 11A8, pH 9.5) in water. The resin was removed by filtration and then stirred in water. The aqueous filtrates (pH 7.3) were combined and concentrated on a rotary evaporator and then dried in vacuo, to yield 64.6 g of material. To remove material bound to the resin, the resin was washed twice with 0.5M ammonium acetate to yield 31.5 g of material, and then with 0.05M acetic acid to yield 9 g of material. The total recovery was 61%.

Burley CEL (190.8 g) was treated with a chelating ion exchange resin (Bio-Rad Chelex 100, pH 5.9 after treatment with sodium acetate buffer, followed by washing with water). Concentration of the resulting aqueous filtrate (pH 5.2) is in progress.

- C. **Plans:** Preparation of fillers and cigarettes will be continued. Cigarettes will be smoked for S/M assay. Samples from the ion exchange treatments will be prepared for spraying. Overhauling and servicing of the electrodialysis unit will be continued. Formulation of plans for C Pilot Plant production of RL will be continued.
- D. **References:**

Hellams, R. Notebook No. 8613, pp. 153-154.
McGee, N. Notebook No. 8743, p. 40.
Drew, S. Notebook No. 8800, pp. 22-23.
Hassam, S. Notebook No. 8712, p. 179.

III. OPTIMIZATION OF A LOW ACTIVITY MODEL: MS NITRIC OXIDE CONTENT

- A. Objective: To develop methods of reducing MS NO delivery for a low activity model.
- B. Results: Two model cigarettes containing an NO oxidant and an NO₂ absorbent were prepared using a dual plug/space/plug filter configuration and all burley filler. The oxidant was a catalyst containing chromium trioxide and potassium dichromate on silica gel. The absorbent was triethanolamine on florisil. The models contained different ratios of oxidant/absorbent. Using current methodology, NO deliveries were determined for these cigarettes. No significant reduction was observed with these cigarettes.
- C. Plans: The use of zeolites as potential catalysts for NO decomposition will be investigated.
- D. Reference:
- Levins, R. J. Notebook No. 8757, p. 172.

IV. DEVELOPMENT AND APPLICATION OF ANALYTICAL PROCEDURES

- A. Objective: To develop, maintain and apply analytical methodology for minor alkaloids and other compound classes.
- B. Results: Twenty-two samples of tobacco filler and eleven ethanol extracts of bright filler obtained from a multiple pass system were analyzed for minor alkaloids and nicotine by capillary GC/NPD.
- C. Reference:
- Levins, R. J. Notebook No. 8757, p. 171.

V. SUPPORT FUNCTION: CONDENSATE PREPARATION

- A. Objective: To fabricate cigarettes, perform smokings, and prepare condensate as needed for biological and chemical analysis.
- B. Results: Three types of fillers were prepared for project 6908: bright base web was sprayed with bright CEL and bright CEL mixed with DAP; burley base web was sprayed with a mixture of bright CEL and burley CEL. Cigarettes were prepared and smoked for other BCR staff. Sixty cigarettes each of four cigarette models were smoked to provide condensate in special plastic containers, for Bob Jenkins (CRD).
- C. References:
- Hellams, R. Notebook No. 8613, p. 153-154.
McGee, N. Notebook No. 8743, p. 40.

PROJECT NUMBER: 8101
PROJECT TITLE: Cigarette Testing Services Division
SECTION LEADER: Jane Y. Lewis
PERIOD COVERED: April, 1989

I. MARKET ACTIVITY

A. Objective: To monitor and report new brand introductions and brand modifications for the domestic and international cigarette markets.

B. Results:

1. Domestic

R. J. Reynolds is distributing Doral Ultra Lights 85 and Magna Lites 80 (Box) cigarettes nationally. The Doral Ultra cigarette delivers 5 mg tar and 0.4 mg nicotine; the Magna Lites 80 (Box) cigarette delivers 9 mg tar and 0.6 mg nicotine. Both of these new value entry products are extensions of their respective brand families.

Lorillard is test marketing Spring Lemon Lights 85 and 100 (plain and menthol) cigarettes in Connecticut. The 85 mm cigarette delivers 9 mg tar and 0.7 mg nicotine; the 100 mm cigarette delivers 11 mg tar and 0.8 mg nicotine. Citral is the source of the lemon flavoring in these products.

The American Tobacco Co. is test marketing American Filter 85 and 100 cigarettes in Illinois. These value entry cigarettes deliver 16 mg tar and 1.1 mg nicotine. American Lights were test marketed by American Tobacco in September, 1988.

II. HUMECTANTS ANALYSIS

A. Objective: To increase efficiency of the gas chromatograph used for humectants analysis.

B. Results: Humectants analyses are performed on a HP5880A GC with 30-meter x 0.53 mm ID DB-WAX megabore columns. The analysis time is about 45 minutes per sample. The installation of a 5-meter x 0.53 mm ID deactivated, uncoated fused-silica guard column before the analytical column resulted in a decrease in the analysis time to 25 minutes per sample.

C. Plans: The Analytical Methods Manual method for humectants in smoke will be updated to incorporate this modification.

III. FILTRONA SMOKING MACHINE

A. Objective: To install a Filtrona 20-port CO smoking machine and implement its use for routine testing.

- B. Results: Evaluation of the Filtrona 20-port CO smoking machine is complete. Monitor 25 results from the Filtrona were 14.3 mg/cigt. CO. The target value is 14.4 mg/cigt. CO. Data from C.I. brands for three months showed comparable results between the Filtrona and the Phipps and Bird smoking machine.
- C. Plans: The Filtrona will be used for generating CO delivery of routine C.I. samples beginning May, 1989.

IV. DYNAMIC RTD/VENTILATION

- A. Objective: To obtain the capability of generating RTD and ventilation data on cigarettes while smoking.
- B. Results: The instrument was delivered on April 13, 1989. Software modifications are being made to achieve the proper output graphically and numerically. The instrument collects data from each of the five ports sequentially for RTD and ventilation. The data are collected on a per puff and per rod basis. Dynamic profiles are plotted following a smoking run.
- C. Plans: To continue testing this instrument to assure consistent operation using monitor cigarettes.